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EVALUATION OF TURBIDIMETRIC METHODS FOR MONITORING OF ASBESTOS FIBERS IN WATER

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A commercially available turbidimeter, modified by the addition of a magnet, was evaluated for the measurement of asbestos in water. The instrument was used to measure the turbidities of asbestos fiber dispersions in non-boiling still water, as functions of both mass and numerical fiber concentrations, for crocidolite, amosite and chrysotile. Results showed that, for well-characterized water sources, the turbidimeter could be used for routine monitoring of amphibole asbestos fibers at the 1 million fibers per liter (MFL) level, if a simple pre-concentration step was included. Additional modifications would be required before the detection level could be reduced to make the technique useful for monitoring of chrysotile at the 1 MFL level.

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FOREWORD

Nearly every phase of environmental protection depends on a capability to identify and measure specific pollutants in the environment. As part of this Laboratory's research on the occurrence, movement, transformation, impact, and control of environmental contaminants, the Analytical Chemistry Branch develops and assesses new techniques for identifying and measuring chemical constituents of water and soil.

Over the past several years, research sponsored and conducted by the U.S. Environmental Protection Agency has been directed toward the development of improved methods for the analysis of asbestos in water. This research has included efforts to develop less expensive and more rapid methods than electron microscopy, which is the most sensitive and specific technique presently in use. In this report, turbidimetry, a sensitive instrumental technique, is investigated for the routine monitoring of asbestos concentrations in drinking water, treatment plant effluent, and other water sources.

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PREFACE

The analytical method for determination of asbestos in water samples has been progressively improved since the issue in 1976 of the Preliminary Interim Method by the U.S. Environmental Protection Agency's Environmental Research Laboratory in Athens, Georgia. The method is based on filtration of the water sample through a sub-micrometer pore diameter membrane filter, followed by preparation of the filter for examination in a transmission electron microscope. Individual fibers are then identified, measured and counted. The procedure is labor intensive, and the resulting high analytical costs limit the extent to which water sources can be monitored routinely for asbestos fibers. Accordingly, a rapid and inexpensive technique is required for routine monitoring of water sources such as the treated effluent from water filtration plants.

In this program, a commercially-available turbidimeter was modified and the use of turbidity measurements as a means of routine monitoring for asbestos fibers in water was investigated.

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ABSTRACT

The performance of a commercially-available turbidimeter was investigated to determine the lowest turbidity measurement which could be discriminated from that obtained using non-boiling still water. The Sigrist L65 turbidimeter was found to be capable of detecting a change of signal equivalent to 0.002 NTU.

The turbidimeter was used to measure the turbidities of asbestos fiber dispersions in non-boiling still water, as functions of both mass and numerical fiber concentrations, for UICC crocidolite, UICC amosite and Union Carbide purified Calidria chrysotile. It was found that a contribution of 0.004 NTU to the measured turbidity corresponded to fiber concentrations of 5 MFL, 2 MFL and 1000 MFL respectively for crocidolite, amosite and chrysotile. For the fiber size distributions used, these fiber concentrations corresponded to mass concentrations of 1.3 µg/L, 1.3 µg/L and 6.0 µg/L for the three varieties of asbestos.

The turbidimeter was modified by the addition of a magnet, so that fibers in the sample cell could be oriented by a 0.3 T magnetic field. A change in turbidity reading with application of the magnetic field was then measured for the fiber dispersion being examined. The change represented the difference between the intensity of the light scattered by the fibers in random orientations and in the systematic orientations controlled by the magnetic field. This difference signal was therefore fiber-specific. The turbidity reading was found to increase if the fibers adopted orientations parallel to the magnetic field direction, and to decrease if the fibers adopted orientations normal to the magnetic field direction. It was found that in the modified turbidimeter a difference signal equivalent to a turbidity change of 0.004 NTU corresponded to fiber concentrations of 3.5 MFL, 12 MFL and 20000 MFL respectively for crocidolite, amosite and chrysotile. For the fiber size distributions used, these fiber concentrations corresponded to mass concentrations of 0.8 µg/L, 7.5 µg/L and 100 µg/L for the three varieties of asbestos.

The detection levels of the prototype magnetic alignment - light scattering system of Chatfield and Riis were much lower than those of the turbidimeter. These corresponded to fiber concentrations of 0.5 MFL, 0.5 MFL and 2.0 MFL for crocidolite, amosite and chrysotile. For the fiber size distributions used, these were equivalent to mass concentrations of 0.1 µg/L, 0.3 µg/L and 0.01 µg/L for the three varieties of asbestos.

The turbidimeter did not have a detection level low enough to permit direct monitoring of asbestos fibers at the 1 MFL level; accordingly, some

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selective concentration technique is required. It was found that the fibers of high-iron amphiboles such as crocidolite could be concentrated using magnetic separation. Starting with a sample volume of 4 liters, the fibers could be separated and re-dispersed in a smaller volume of clean water. Some variability in the numerical fiber separation efficiency was observed (14% - 43%), but the mass recovery of the technique was nearly constant at about 65%. The technique was shown to be capable of concentrating fibers by at least a factor of ten prior to the turbidity measurement. Selective concentration of chrysotile cannot be achieved by magnetic separation, and other techniques were investigated. The turbidity of drinking water samples could generally be reduced by a factor of ten, using a combination of ozone-ultraviolet light treatment and filtration. It was found that, if Nuclepore filters with pore diameters between 0.8 μm and 3.0 μm were used for the filtration step, most of the chrysotile fibers of sizes normally found in drinking water would pass through. Using ozone-UV treatment and filtration through a 2.0 μm pore diameter filter, the turbidity of a drinking water sample was reduced from 0.109 NTU to 0.0165 NTU, and 78% of the chrysotile fibers passed through into the filtrate. This simple technique yields a net concentration of fibers relative to other sources of turbidity.

The scavenging of chrysotile fibers by container surfaces reported by Chatfield et al was investigated further, with a view to developing it as a fiber separation technique. It was found that, for unknown reasons, the procedure was not so effective as it had been in the previous studies, and that erratic separation efficiencies of less than 20% were obtained. It was concluded that a complete investigation into the mechanism of this effect was required before it could be applied routinely.

It was concluded that for well-characterized water sources the turbidimeter could be used for routine monitoring of amphibole asbestos fibers at the 1 MFL level, if combined with a simple pre-concentration step. Further modifications of the instrument would be required before the detection level could be reduced to make the technique useful for monitoring of chrysotile at the 1 MFL level. The prototype magnetic alignment - light scattering system, in some cases directly, but in others with the requirement for simple pre-concentration steps, could be used for routine monitoring of all asbestos fiber varieties at the 1 MFL level.

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LIST OF ABBREVIATIONS

AWWA - American Water Works Association

EPA - U.S. Environmental Protection Agency

FNU - Formazin Nephelometric Unit

JTU - Jackson Turbidity Unit

kPa - kilopascals

L - liter

µg - micrograms (10^{-6} grams)

MFL - Million Fibers per Liter

mL - milliliter (10^{-3} liter)

NTU - Nephelometric Turbidity Unit

ppm - parts per million

SiO_2 - silicon dioxide

T - Tesla

TEM - transmission electron microscope

UICC - Union Internationale Contre le Cancer

UV - ultraviolet

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SECTION 1

INTRODUCTION

Measurements of asbestos fiber concentrations in water are made using transmission electron microscopy (TEM). As a first step in establishing a satisfactory analytical method, a preliminary version of an interim procedure was published by the EPA in 1976.[1] A revised version, incorporating some improvements, was published in 1980.[2] In 1983, a further development of the method was published which incorporated sample conditioning steps and precise definition of fiber identification procedures.[3]

Analyses using the TEM method are time-consuming and expensive, because each sample requires examination in an analytical electron microscope for at least three hours, during which every fiber is separately classified and measured. Satisfactory analyses require that the microscope operators have a high degree of skill and experience.

The high cost and the long turnaround time of analyses made by electron microscopy have prompted investigations into whether more rapid analytical procedures could be developed, even if these were not capable of comparable specificity and sensitivity. The limitations of several potential technical approaches to this problem were discussed by Chatfield and Riis[4]. They concluded that the major cost of current analyses was a result of the labor expended in manual fiber counting, and that a rapid method could not incorporate this in any form. The technique they selected for development was based on optical scattering by magnetically-aligned fiber dispersions, a phenomenon first reported by Timbrell[5]. Before the work of Chatfield and Riis, another technique had been developed by Diehl et al[6] in which asymmetric optical scattering by single fibers, as they passed through a focused laser beam, was detected by an array of photomultipliers. However, both of these instrumental techniques are at the prototype stage. Accordingly, there is interest in the concept of using established instrumental techniques, perhaps with minor modifications, to monitor asbestos fiber concentrations in water. Turbidimetry is a sensitive technique which has been applied by several investigators to the problem of measurement of asbestos in drinking water. However, in summary it appears that two apparently contradictory conclusions were reached:

(a) turbidity readings can be used as an indicator of fiber removal efficiency in a filtration plant;[7,8]

(b) no definite correlation between fiber concentration and turbidity was found.[9,10,11,12]

The situation is further complicated by the fact that most of the fiber concentration values reported in these studies of turbidity would now be considered questionable because of the methods used in the preparation of specimens for the transmission electron microscope evaluations. For example, the fiber concentration - turbidity relationship derived by Lawrence and Zimmermann[10] for "pure" chrysotile dispersions was based on TEM measurements which used specimens prepared by centrifugation to concentrate the suspension, followed by micropipetting of the concentrate onto carbon-coated grids. This preparation technique is no longer considered reliable for quantitative determinations. Another factor which should be considered is that all of the data points in this study were derived by successive dilution of a single chrysotile dispersion, the concentration of which was open to question. The products of biological activity are now known to de-stabilize aqueous dispersions of chrysotile fibers.[13] Therefore, the validity of this work, and also the other studies based on it, [8-12] would be questionable because of the unsterile conditions used in handling of the chrysotile dispersions. Further support for this view is found in the obvious inconsistencies which exist between the mass of chrysotile used to prepare the dispersion and the numerical fiber concentration reported. It is therefore probable that the results of these studies were much more seriously affected by the inability to prepare stable fiber dispersions, than they were by inadequacies of the fiber counting technique in use at the time. Indeed, the authors commented on inconsistent results from what were considered to be dispersions with known fiber concentrations.[11]

Clearly, there is no reason why the asbestos fiber concentration should be related to the turbidity measurement, unless the asbestos is the principal particle species which gives rise to the turbidity in the particular water sample. This is unlikely to be the case in most water systems. Nevertheless, there are two ways in which turbidity measurements may be useful in monitoring asbestos fiber concentrations: firstly, if the fibers could be concentrated selectively so that they form the major contribution to the turbidity measurement; and secondly, if the turbidity of the sample could be reduced so that the residual value corresponds to a fiber concentration below some level of concern. There would also be an advantage if the turbidity measurement itself could discriminate between the contributions made by fibrous and equant particles. In this current study, all of these aspects have been investigated to determine whether measurement of turbidity could provide a realistic method for monitoring asbestos fiber concentrations in drinking water.

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SECTION 2

CONCLUSIONS

The experiments showed that the Sigrist L65 turbidimeter was capable of detecting a turbidity increment of 0.002 NTU above the value for non-boiling still water. Other turbidimeters were not able to achieve this detection level. The Sigrist L65 turbidimeter employs stable standards calibrated in terms of silica, and it was found that an ambiguity exists in the published calibration procedure against formazin standards, if such calibrations are extended to values below about 0.1 NTU. This ambiguity is a consequence of the turbidity of water used for dilution of the formazin reference standard. In the published procedure, this is referred to as "turbidity-free water", and at levels below 0.1 NTU it can no longer be considered to be "turbidity-free".

Relationships between turbidity and asbestos fiber concentration were obtained for UICC crocidolite, UICC amosite and Union Carbide refined Calidria chrysotile. These indicated that a contribution of 0.004 NTU to the total turbidity corresponded to asbestos fiber concentrations of 5 MFL, 2 MFL and 1000 MFL for UICC crocidolite, UICC amosite and Union Carbide chrysotile respectively. These corresponded to mass concentrations of about 1.3 µg/L, 1.3 µg/L and 6.0 µg/L respectively for the three varieties of asbestos.

A modification was made to the turbidimeter which allowed the sample to be placed between the poles of an electromagnet. The magnetic field caused the fibers to become oriented, and it was possible to observe a change in the turbidity reading when the field was applied. A change of 0.004 NTU in the turbidity reading corresponded to fiber concentrations of 3.5 MFL, 12 MFL and 20,000 MFL for UICC crocidolite, UICC amosite and Union Carbide chrysotile respectively. These corresponded to mass concentrations of 0.8 µg/L, 7.5 µg/L and 100 µg/L respectively for the three varieties of asbestos. In the modified turbidimeter, with application of the magnetic field, fibers which adopted an orientation parallel with the field (p-fibers) caused an increase in the turbidity reading; fibers which adopted orientations normal to the field (n-fibers) caused a reduction in the turbidity reading. Increases in the turbidity readings were obtained using crocidolite, amosite, chrysotile, anthophyllite and cummingtonite. Decreases were observed using tremolite and actinolite. In the case of amosite, the signal from the two alignment modes of fiber partially cancelled, and different magnitudes of effect were obtained with two different varieties of amosite. Theoretically, some fibrous minerals or mixtures of fibers could exist in which the effects from the two alignment

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modes of fiber completely cancel, leading to an incorrect conclusion that no fibers are present. The detection levels of the magnetic alignment - light scattering system of Chatfield and Riis[4] were lower than those for either configuration of the turbidimeter, and these were achieved using a 5 mL sample, rather than the 65 mL or 130 mL required by the turbidimeter. The detection levels were 0.5 MFL, 0.5 MFL and 2 MFL respectively, for crocidolite, amosite and chrysotile, corresponding to mass concentrations of 0.1 µg/L, 0.3 µg/L and 0.01 µg/L. This system also does not suffer from the signal cancellation effect observed in the turbidimeter for varieties containing fibers of both alignment modes.

It was concluded that the detection levels of both the modified turbidimeter and the magnetic alignment - light scattering system could be improved by use of more sophisticated time averaging and read-out techniques.

It was found that magnetic separation was an effective procedure for concentration of crocidolite from water samples. Starting with 4 liter water samples, the amphibole fibers could be separated and transferred to a 65 mL water sample for the turbidimeter. The numerical fiber recovery was variable for some reason, between 14% and 43%, although the mass recovery was constant at about 65%, indicating that the larger fibers were recovered more reproducibly. Concentration of the fibers by factors of between 8.7 and 26.9 were achieved, equivalent to a factor of 40 in terms of mass concentration. Magnetic separation cannot be used for concentration of chrysotile and low-iron amphiboles, and other methods must be used.

In order to improve detection limits, techniques were investigated to reduce the turbidity of the water sample, while retaining the majority of the asbestos fibers. Filtration of the sample through large pore diameter Nuclepore filters was studied as a means to separate the asbestos fibers from other particulate material on the basis of differences in their size distributions. It was shown that for a typical size distribution of waterborne chrysotile fibers, Nuclepore filters of pore diameters exceeding 0.8 µm allowed most of the chrysotile asbestos fibers of an artificial dispersion to pass through. This was demonstrated using 0.8 µm, 2.0 µm and 3.0 µm pore diameter filters. After such a filtration, the turbidities of typical drinking water samples were significantly reduced, usually by a factor of about 10 when 0.8 µm pore diameter filters were used. The technique was more effective for samples of drinking water if the organic materials were oxidized prior to this selective filtration step, indicating that many of the fibers were associated with organic materials in the original drinking water sample. Using a 2.0 µm pore diameter filter, 78% of the fibers in an artificial dispersion prepared using a drinking water sample were found to pass through to the filtrate, and at the same time the turbidity was reduced from 0.109 NTU to 0.0165 NTU. Therefore, this procedure would achieve an improvement of about a factor of 5 in the detection limit.

The fiber entrapment procedure based on the observations of Chatfield et al[13] was investigated further. It was found that the separation

efficiency was in no case more than 20%, and that increase in the surface area of plastic exposed to the fiber dispersion did not increase the efficiency. It was concluded that some aspects of this procedure were not sufficiently understood, and development of the technique to a useful point was not possible within the constraints of this investigation.

It was concluded that the turbidity measurement made by the modified instrument, with prior concentration of the fibers by magnetic separation, would allow fiber-specific monitoring of high-iron amphiboles at the 1 MFL level. The utility of the instrument for low-iron amphiboles depends on the extent to which the detection level can be improved by signal averaging, and on the effectiveness of the selective filtration technique for amphibole fibers. Further modification of the instrument would be required before it could be used to monitor chrysotile at realistic concentrations. The magnetic alignment - light scattering system, with only a simple pre-concentration step, could be used to monitor the concentration of any fiber variety at concentrations less than 1 MFL.

SECTION 3

RECOMMENDATIONS

It is recommended that time averaging and digital output techniques be applied to improve the detection limits of the modified turbidimeter.

More work should be conducted on improvement of the modified turbidimeter for detection of chrysotile asbestos. It is not yet clear whether a magnetic field of 0.3 T was sufficient to achieve satisfactory fiber alignment in the case of chrysotile.

Further modifications of the turbidimeter should be investigated, in order to optimize its performance. In particular, the use of a monochromatic or laser light source and the effects of conversion to conform with the ISO Draft International Standard should be investigated.

Experiments should be conducted to determine the range of iron content in amphibole fibers for which magnetic separation is useful. This work would be relevant not only as a concentration step prior to measuring samples using the turbidimeter, but also in many other areas of asbestos analysis.

Experiments should be conducted to improve the design of the simple magnetic separator, so that higher liquid flowrates can be used without reducing the collection efficiency.

More work should be carried out to determine the mechanism by which chrysotile asbestos fibers are scavenged by the inside surfaces of plastic containers. This effect has implications for the removal of asbestos from water sources. The agents which promote the effect have not yet been precisely identified, and all of the factors controlling the mechanism are not understood.

Further work should be conducted to optimize the reduction of turbidity from non-fibrous particles in drinking water samples prior to measurement for fibers.

More work should be carried out to determine the effect of fiber size distribution on the turbidity measurement.

SECTION 4

PRINCIPLES OF TURBIDITY MEASUREMENT

Turbidity in water is caused by the presence of suspended matter such as clay, silt, finely divided organic and inorganic matter, plankton and other microscopic organisms. Turbidity is an expression of the optical property that causes light to be scattered and absorbed, rather than transmitted in straight lines through the water.

Turbidity was once measured using the Jackson candle turbidimeter. The principle of operation was to view the flame of a standard candle, which was burning at a specified rate, through a container of the liquid. The candle flame would become obscured by the background scattered light when viewed through a sufficient depth of liquid, and a turbidity measurement was made by recording this depth. The turbidity value in Jackson Turbidity Units (JTU) was then obtained from calibration tables. This technique was not practical for measurements on clearer liquids having turbidities below 25 units on the Jackson turbidity scale. Treated water samples have values which, on this scale, would approximate to between 0 and 5 units.

Commercial instruments now available are capable of measuring low turbidities and are based on measurement of the intensity of light scattered by the liquid at some angle to the incident beam direction. Most instruments measure the light scattered at 90° to the incident direction, although a few measure the forward scattering at 15°. Measurement of 90° scattering is less sensitive to the presence of large particles than is measurement at 15°.[14] The light scattered by the unknown sample is compared with that scattered by a reference standard. The standard usually used, and specified by the EPA,[15] is a freshly-prepared reference suspension of the polymer formazin, prepared by mixing solutions of hydrazine sulfate and hexamethylenetetramine under closely controlled conditions. The scattered light measurement is specified in nephelometric turbidity units (NTU). Most data are now reported in terms of this unit, though the alternative designation of formazin turbidity unit (FTU) is often used. A Draft International Standard for determination of turbidity (approved by the USA in Proposal form) has been published by the International Organization for Standardization (ISO), in which the ambiguity of terminology has been resolved by defining the formazin nephelometric unit (FNU).[16]

Because there is no direct relationship between the intensity of light scattered at 90° and the Jackson candle turbidity, calibration of the nephelometric type of instrument in terms of Jackson turbidity units has no valid basis, and the two sets of units are considered comparable but not

equal. This practice is not always followed in the literature, and values below 1 JTU have been specified,[17,18] when by definition the JTU scale does not extend below 25 units.

There is some difficulty in comparing results obtained using different instruments. The Draft International Standard[16] specifies the measurement geometry and conditions required if instrument-to-instrument comparisons are to be valid. Figure 1 illustrates this geometry, and the following conditions apply:

(a) the wavelength of the incident radiation shall be 860 nm;

(b) the bandwidth of the incident radiation shall be less than or equal to 60 nm;

(c) there shall be no divergence from parallelism of the incident radiation, and any convergence shall not exceed 1.5°;

(d) the measuring angle θ, between the optical axis of the incident radiation and that of the scattered radiation shall be 90 ±2.5°;

(e) the aperture angle ω_o , shall be between 20° and 30°.

There is no way to avoid the occurrence of stray light in a turbidity measurement instrument. Light is scattered by the internal optical components and by the sample cell, so that a definite instrumental reading is obtained even without a contribution from the sample itself. The determination of stray light in a particular instrument is not simple, and if accurate measurements below 0.1 NTU are to be made, the value must be known. It cannot be determined simply by the use of distilled water as a sample, because even distilled water is not free of turbidity.

The procedure for turbidity measurements in the ranges below 0.1 NTU requires a great deal of care. The cells must be very clean, air bubbles in the sample must be eliminated and smoke or dust in the laboratory must be minimized.

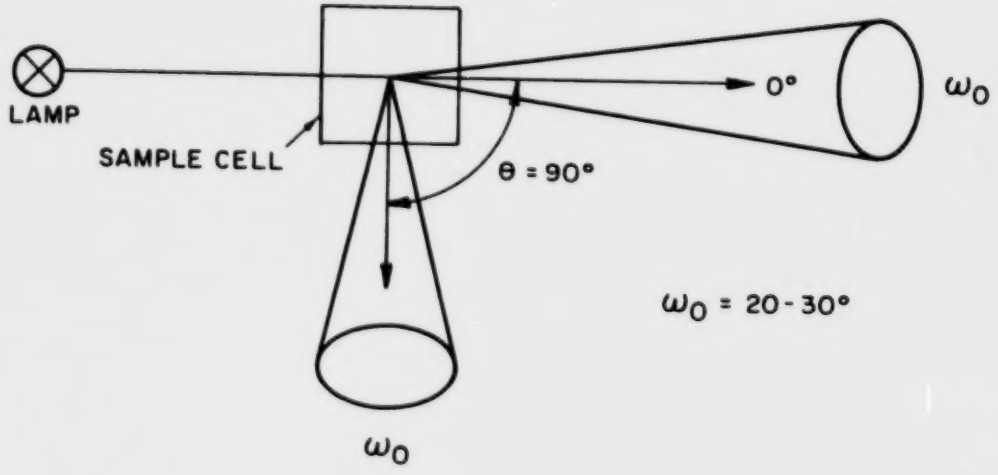

Figure 1. Measurement Geometry Required if Comparisons Between Results Obtained on Different Instruments are to be Valid.

SECTION 5

EXPERIMENTAL PROCEDURES

INSTRUMENT EVALUATION AND MODIFICATION

Evaluation of the Measurement Problem

In order to define the work program, it was necessary to obtain order of magnitude estimates of the contributions from pure asbestos fiber dispersions to routine turbidity measurements, and to compare these with turbidity levels obtained on municipal drinking waters. Three initial measurements were made using a Hach Model 2100A turbidimeter. The values obtained are summarized in Table 1. It can be seen that after deduction of the 0.04 NTU for the blank, an equivalent of 0.02 NTU is to be compared with a chrysotile concentration of 200 MFL, rising to 0.04 NTU at a chrysotile concentration of about 1000 MFL. About 140 MFL of crocidolite appears to be equivalent to a reading of 0.07 NTU. Turbidity readings on treated water supplies studied in a Canadian survey[19] ranged from 0.04 NTU to 1.5 NTU, which does not conflict with the observation that U.S. values fall within the range of 0 to 5 NTU.[14]

TABLE 1. INITIAL TURBIDITY MEASUREMENTS MADE ON ASBESTOS FIBER DISPERSIONS

Sample	Fiber Concentration		Turbidity
	MFL*	µg/L	NTU
Double-Distilled Water	< 0.01	< 0.001	0.04
Union Carbide Chrysotile	1000	0.5	0.08
Union Carbide Chrysotile	200	0.1	0.06
UICC Crocidolite	140	50	0.11

*Million Fibers per Liter

These initial measurements indicate that detection of 1 MFL of chrysotile in otherwise turbidity-free water would correspond to about 0.0001 NTU. In water which meets the quality goal of about 0.1 NTU for

turbidity, the contribution from 1 MFL of chrysotile would be about 0.1% of the total measured value. However, treated water turbidities up to about 5 NTU are often encountered, which aggravates the measurement problem still further.

It is certain that because of stray light problems, commercially available instruments currently do not have the capability of measurement down to 0.0001 NTU, and they certainly do not permit readings to a precision of 1 part in 50,000. Use of a turbidity measurement as an indication of asbestos fiber concentration will require that:

(a) the sensitivity of the instrument to very low turbidity values be improved; and

(b) the total turbidity of a grab sample be reduced by simple pre-treatments such that the contribution from any asbestos fibers becomes a higher proportion of the total measurement.

An alternative approach would be to modify the turbidimeter so that its response to fibers is different from that to other shapes of particle. This feature could be incorporated by alignment of the fibers, either by a magnetic field or by a fluid velocity gradient.

Selection of Turbidimeter and Calibration

Table 2 shows the turbidimeters selected for evaluation in this program, the sources of supply, and the most sensitive range on each instrument. Some of the instruments available do not provide ranges below 0 - 1 NTU, and these were not considered further. Waterborne asbestos fibers are usually very small, therefore the type of turbidimeter which measures light scattered at 90° to the direction of the incident beam was considered to be the most suitable, in view of the relative insensitivity of this design to the presence of large particles.[14] All of the instruments listed in Table 2 are of this type. One instrument manufactured by Monitek Inc., 1495 Zephyr Avenue, Hayward, California 94544, was eliminated from further consideration because its design was based on measurement of forward scattering.

It was found that the basic designs of the DRT-100, Hach 2100A and DRT-280 were very similar, and only the Hach 2100A was evaluated further. The Hach 18900 was different, in that it incorporated a ratio measurement technique using the incident beam as a reference. The design of the Sigrist L65 is different from those of other instruments, in that it uses the null principle and fixed reference standards.

A Hach 2100A turbidimeter was cleaned and set up according to the manufacturer's instructions. The reference standards provided with the instrument were used for calibration, after which a series of reference formazin standards were prepared[15] and measured. The results obtained are shown in Table 3. This instrument yielded a value of 0.03 NTU for double-distilled water. However, the most serious problem was one of instability

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TABLE 2. TURBIDIMETERS SELECTED FOR EVALUATION

Model	Manufacturer	Maximum Sensitivity Range (NTU)
DRT-100	H-F Instruments Division Shaban Manufacturing Inc. 3052 Hardee Street Fort Myers Florida 33901	0 - 0.1
Hach 2100A	Hach Company P.O. Box 389 Loveland Colorado 80537	0 - 0.2
DRT-280	Lisle-Metrix Ltd. 49 Sheffield Street Toronto, Ontario Canada M6M 3E5	0 - 0.1
L65	Sigrist-Photometer Ltd. Zweierstrasse 129 Ch-8036 Zurich Switzerland	0 - 0.1
18900	Hach Company P.O. Box 389 Loveland Colorado 80537	0 - 2 (Digital)

when attempting to measure samples with turbidities lower than 0.2 NTU. The compact design was also a disadvantage for the purposes of this study, in which it was intended to make modifications to the instrument.

TABLE 3. EVALUATION OF HACH 2100A TURBIDIMETER

Reference Formazin Sample, NTU	Reading of Hach 2100A, NTU
400	420
100	92
10	6.2
1	1.02
0.2	0.26
0.1	0.14
0.05	0.10
Double-Distilled Water	0.03

The Hach 18900 Ratio Turbidimeter was found to have significantly better stability than the 2100A for measurement of low turbidity samples. However, the stray light signal appeared to be comparable with that of the 2100A, and a turbidity measurement on double-distilled water yielded a value of 0.02 NTU. Like the 2100A instrument, this instrument did not provide a resolution better than 0.01 NTU.

The Sigrist L65 turbidimeter operates on a null principle, illustrated in Figure 2. The oscillating mirror (M) is driven by an alternating power supply (A). Light from the bulb (L) is reflected by the oscillating mirror (M) alternately into two paths. Light along one path is reflected by the mirror (R) into the sample cell (C), and scattered light at 90° from this beam direction is detected by the photocell (P). Light along the other path passes through a variable density wedge (W) and a turbidity reference standard (S) into the cell, and then straight on to be detected by the photocell. The variable density wedge is adjusted until no alternating signal is detected by the photocell and lock-in amplifier (B), the null point being indicated on a meter (N). The turbidity measurement is then made by observing the position of the variable density wedge, indicated on a calibrated drum (I). The drum is calibrated in percent (of the reference standard value). The stray light value on the Sigrist L65 can be obtained by a specified procedure. The reading on the drum can be obtained with a

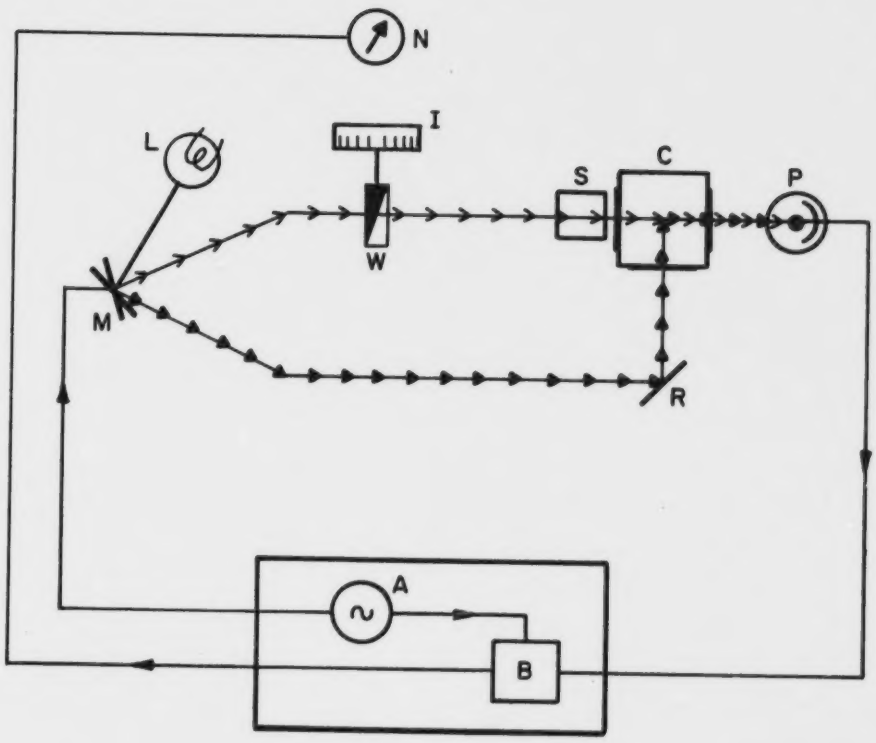

Figure 2. Operating Principle of the Sigrist L65 Turbidimeter.

resolution of 0.5% of the reference standard in use. The lowest reference standard of 0.5 mg/L SiO_2 corresponds to approximately 0.1 NTU, leading to a resolution of about 0.0005 NTU. While it is recognized that a single measurement cannot be made to an accuracy of 0.0005 NTU, the reading on the instrument for a sample of double-distilled water was stable to within 0.001 NTU.

In view of the high resolution and stability requirements of the program, the Sigrist L65 was selected as the instrument which most closely met these criteria.

Formazin turbidity standards were prepared, and the instrument was calibrated. Figures 3 and 4 show the calibrations obtained; this procedure was repeated at intervals during the program. Using the 0.5 mg/L SiO_2 standard, it was found that 1 scale division corresponded to 0.00165 NTU. Thus, within the limits of resolution, the detection limit was approximately 0.002 NTU when using this standard.

Turbidities of Asbestos Fiber Dispersions

The possible use of turbidity measurements as an indicator of asbestos fiber concentrations requires an accurate knowledge of the contribution made to the turbidity measurement by specific concentrations of fibers which have size distributions similar to those observed in drinking water. The stable reference dispersions of asbestos fibers developed in the previous work[13], and new dispersions of higher concentrations, were used to measure the fiber concentration - turbidity relationships for UICC crocidolite, UICC amosite and Union Carbide refined Calidria chrysotile. The size distributions of these reference dispersions are shown in Appendix A.

Dispersions with a range of fiber concentrations were prepared by successive dilution of the reference dispersion using "turbidity-free" water. Previous work using the magnetic alignment system had demonstrated the extreme difficulty in producing particle-free water, and that even double-distilled water from a glass still contained sufficient numbers of particles to cause some interference.[4] In view of this experience, it was decided that a quartz non-boiling still should be used to produce the high quality, particle-free water required for this project. The principal source of particles in distilled water is from rapid boiling of the source water, which produces spray when the bubbles burst. The spray is carried over into the distilled product. In the non-boiling still, the water is heated from above by an infra-red heater, and distillation occurs by evaporation at temperatures below the boiling point. The non-boiling still, therefore, is not subject to carry-over of particles from the source water. A suitable still was the model PB5, manufactured by Quartz et Silice, Les Miroirs (Defense 3), Cedex 27-92096 Paris La Defense, France, which is capable of producing about 500 mL per hour.

The results obtained for measurement of the turbidity of different fiber concentrations of UICC crocidolite are shown in Figure 5. When this work was initially performed, it was immediately evident that

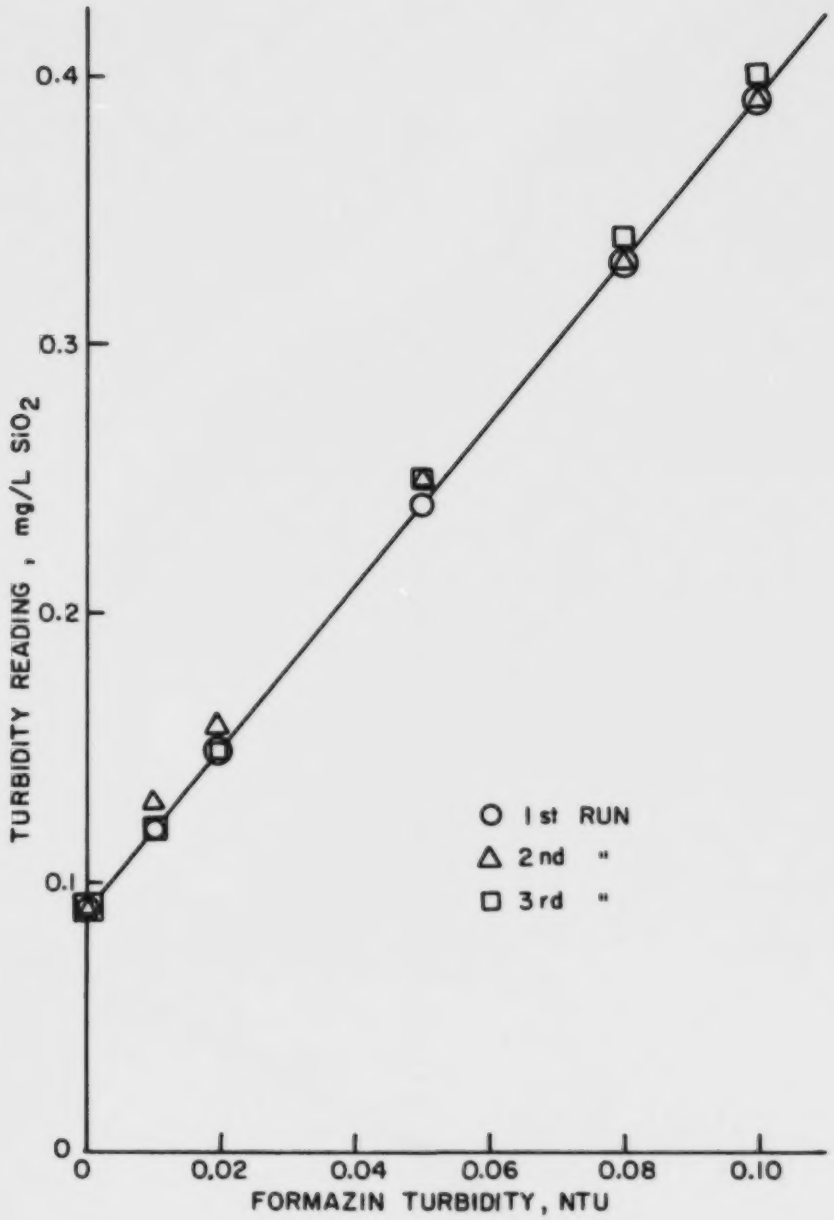

Figure 3. Calibration of Turbidimeter Against Formazin Standards, 0-0.1 NTU for 0.5 ppm SiO_2 Reference Standard.

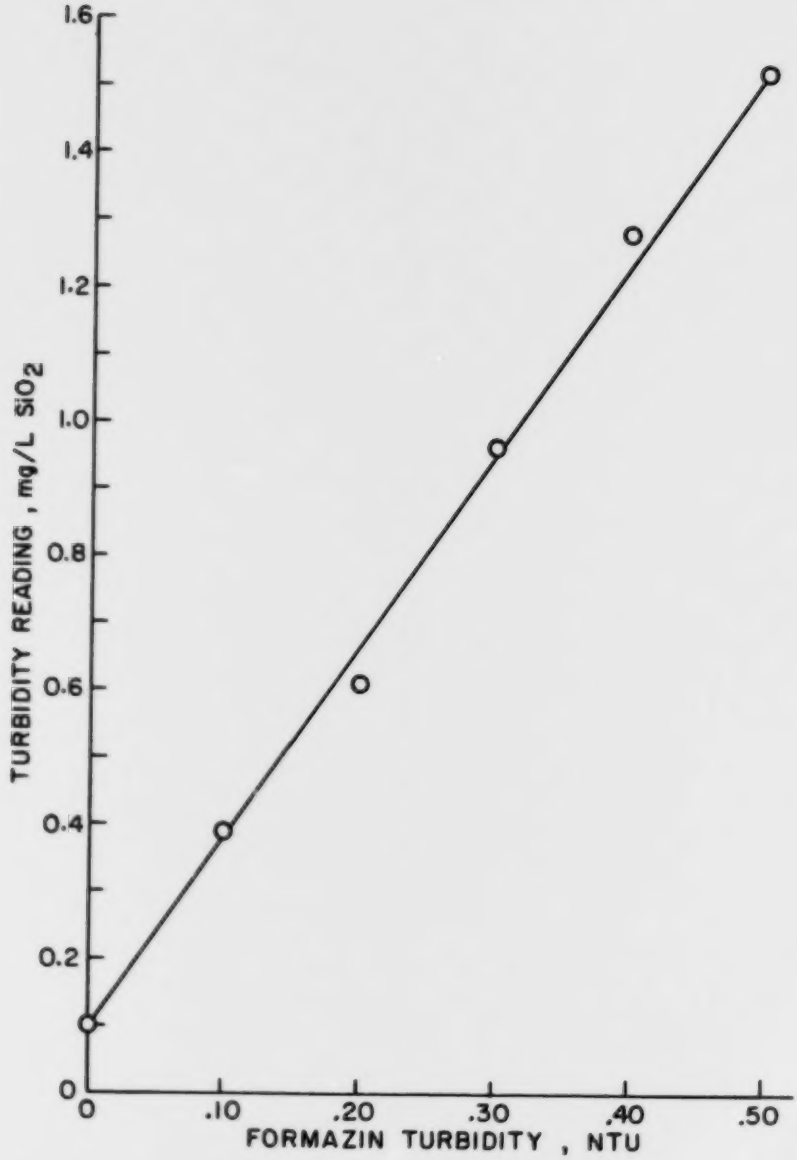

Figure 4. Calibration of Turbidimeter Against Formazin Standards, 0-0.5 NTU for 2.0 ppm SiO_2 Reference Standard.

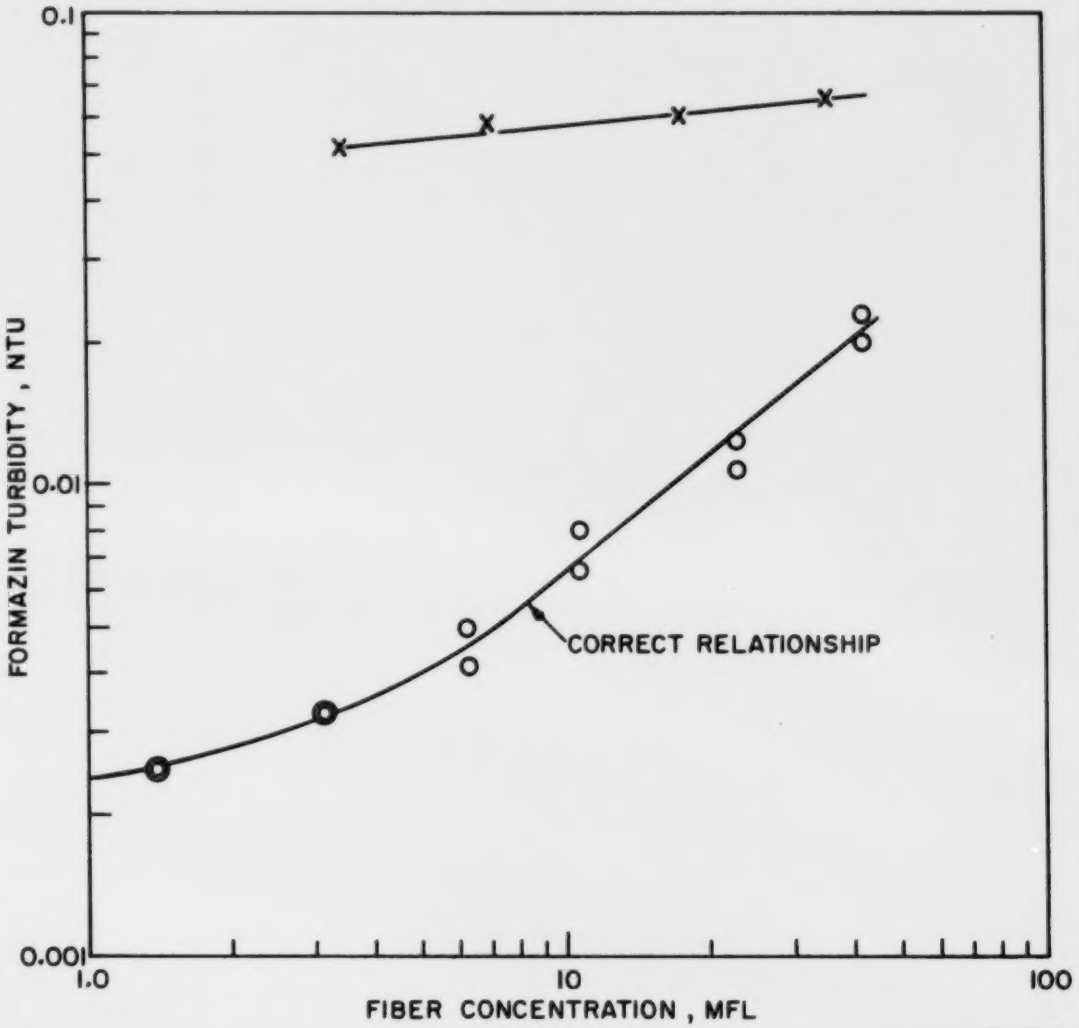

Figure 5. Turbidity of UICC Crocidolite Asbestos Dispersions.

measurement of turbidities below 0.1 NTU is a procedure requiring a great deal of skill. Particles of dust, airborne smoke, and dirt on either the optical components or the sample cell itself are capable of introducing very large relative errors into the measurement. The relationship denoted by crosses in Figure 5 was obtained when insufficient attention had been paid to cleanliness. As would be expected, the correct relationship approaches the detection limit of 0.002 NTU asymptotically for low fiber concentrations. Two calibrations were made, starting out from two separate ampoules of the reference dispersion. The majority of the measurements representing the correct relationship are below the turbidity equivalent of the background value (0.0297 NTU). For each sample, four measurements should be made, rotating the cell by 90° between measurements. Any significant difference between these readings is cause for the measurements to be rejected and repeated after thorough cleaning of the sample cell. For precise work, the readings used should all be taken with the cell in a fixed orientation and using the same cell.

The results for UICC amosite are shown in Figure 6. A similar pattern of results was obtained, although the precise relationship was not the same as that for crocidolite, probably because of differences in the fiber size distributions.

The data for Union Carbide chrysotile are shown in Figure 7, in which a curve obtained by Lawrence and Zimmermann[10] is also shown for comparison. The chrysotile fiber concentration - turbidity relationship reported by Lawrence and Zimmermann is clearly in error, because the turbidity value found at low fiber concentrations approached an asymptotic value of about 0.3 NTU, compared with a measurement of about 0.04 NTU which should have been possible for turbidity-free water even by use of the simplest turbidimeters. In published work on the use of turbidity measurements to determine asbestos fiber concentrations, however, the Lawrence and Zimmermann study appears to be the only one in which an attempt was made to determine a fiber concentration - turbidity relationship.

In Figure 8, the turbidity measurement appears to be the same function of mass concentration in the case of crocidolite and amosite. However, the relationship is different for chrysotile, probably because the diameters of the chrysotile fibers were much smaller than those of the amphibole varieties.

Modification of Turbidimeter

A modification was made to the turbidimeter which permitted asbestos fibers to be held aligned by a magnetic field while the turbidity measurement was made. Figure 9 shows the magnet assembly, which was designed to accommodate the sample cell in the normal position. All components of the magnet assembly were painted in a matte black to eliminate possible reflections of stray light. It was found possible to reduce the sample volume from the standard 130 mL to 65 mL without affecting the performance of the instrument; this allowed the upper pole piece of the magnet to remain above the surface of the liquid in the sample cell. An increase in the

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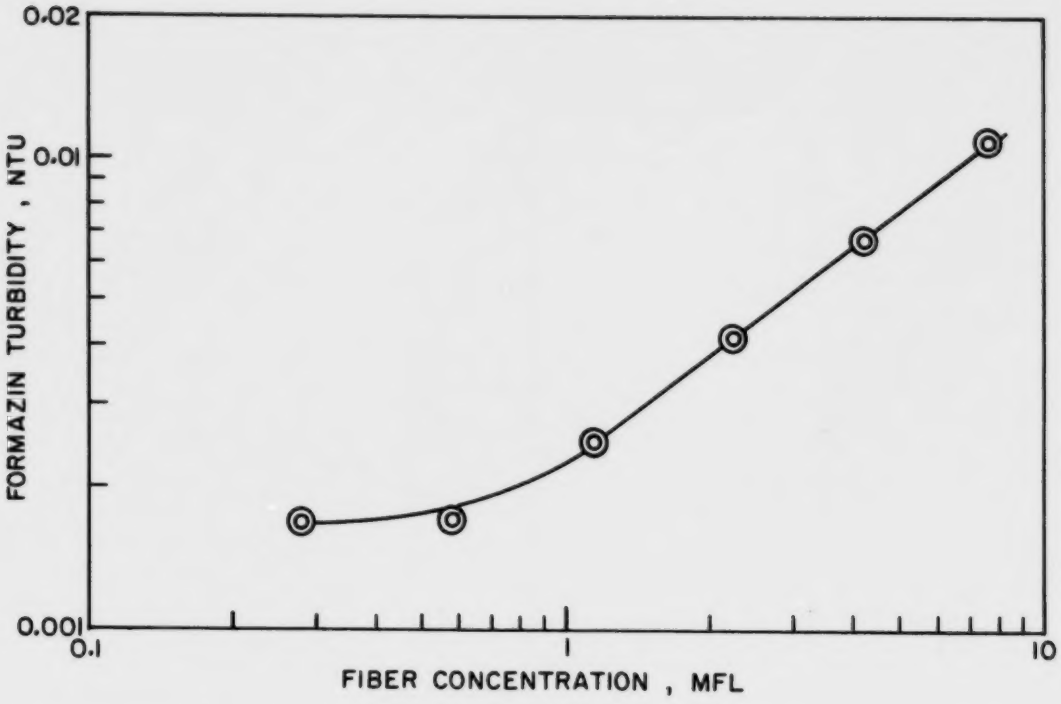

Figure 6. Turbidity of UICC Amosite Asbestos Dispersions.

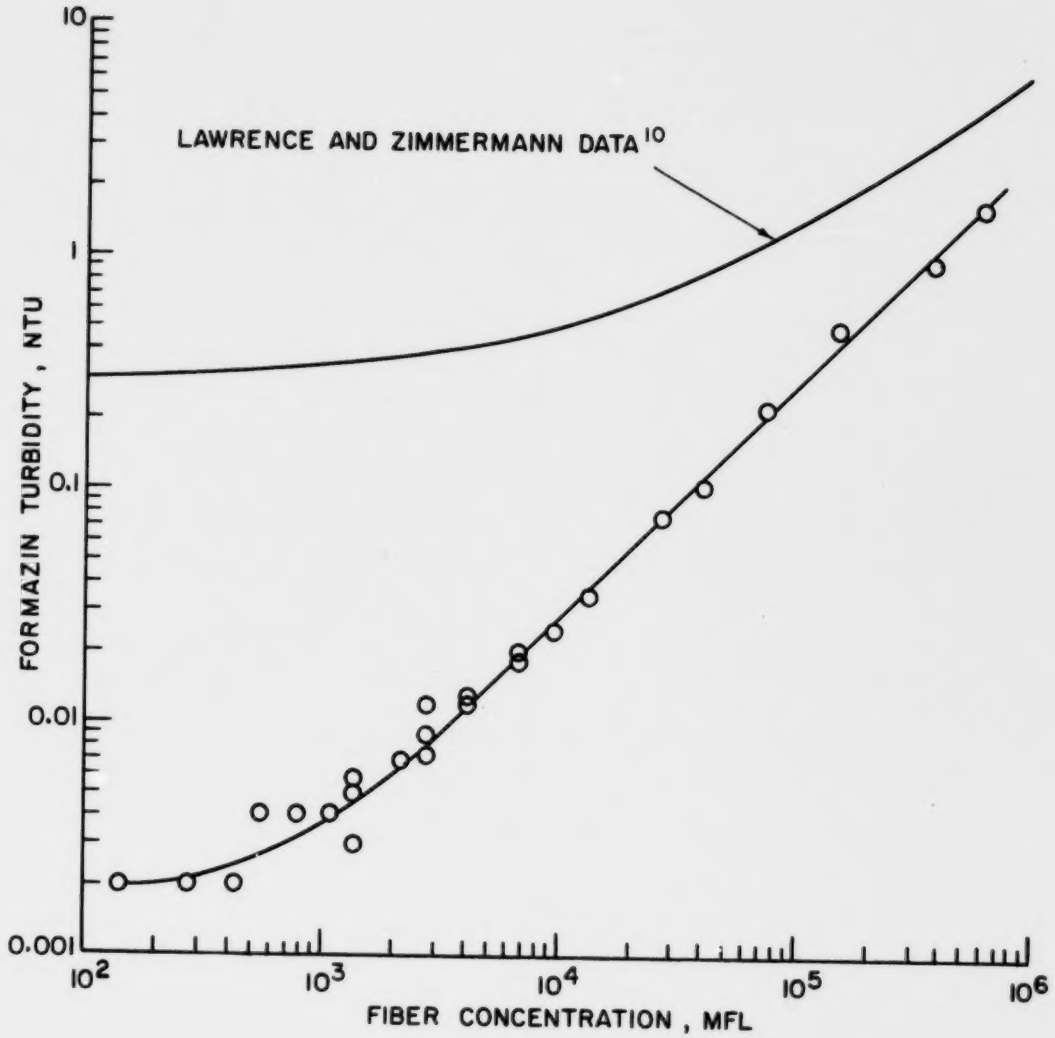

Figure 7. Turbidity of Union Carbide Chrysotile Asbestos Dispersions.

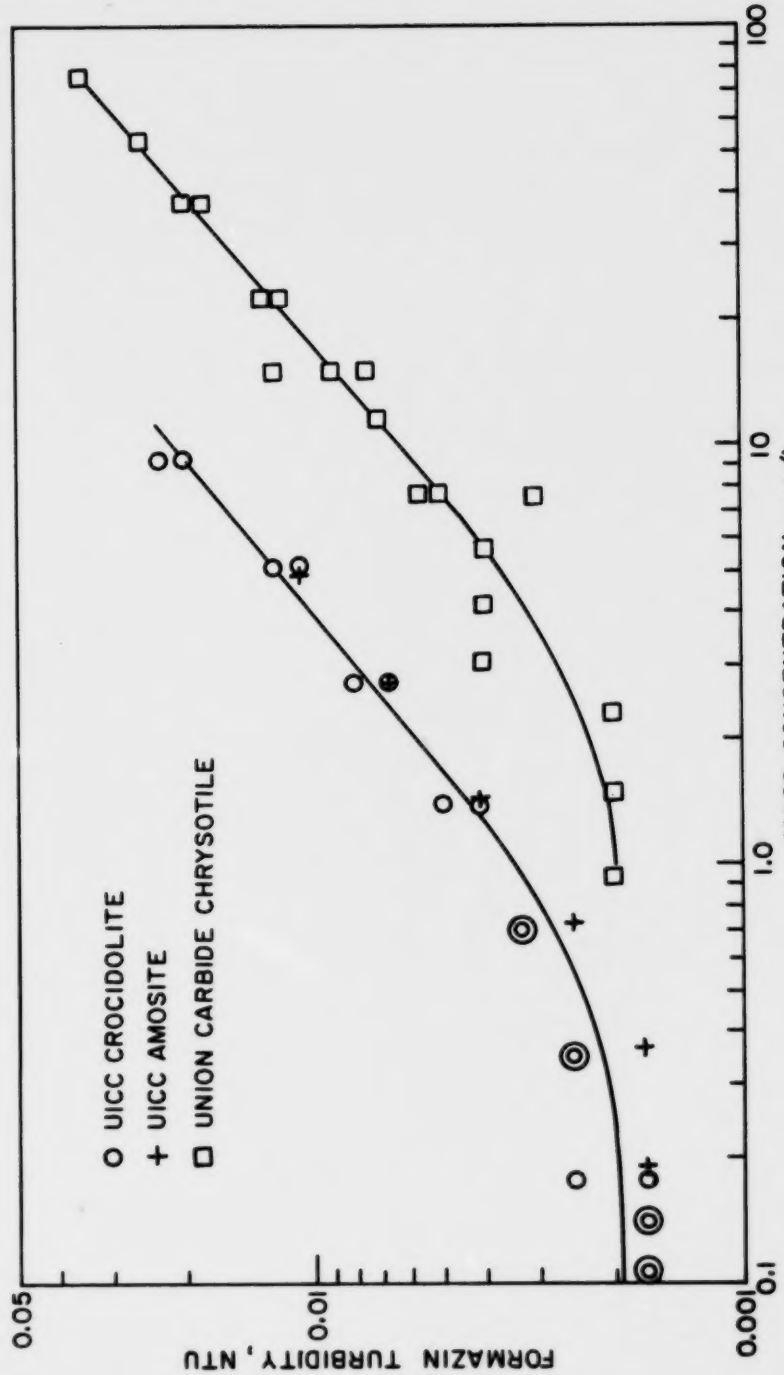

Figure 8. Formazin Turbidity vs Mass Concentration for UICC Crocidolite, UICC Amosite and Union Carbide Chrysotile.

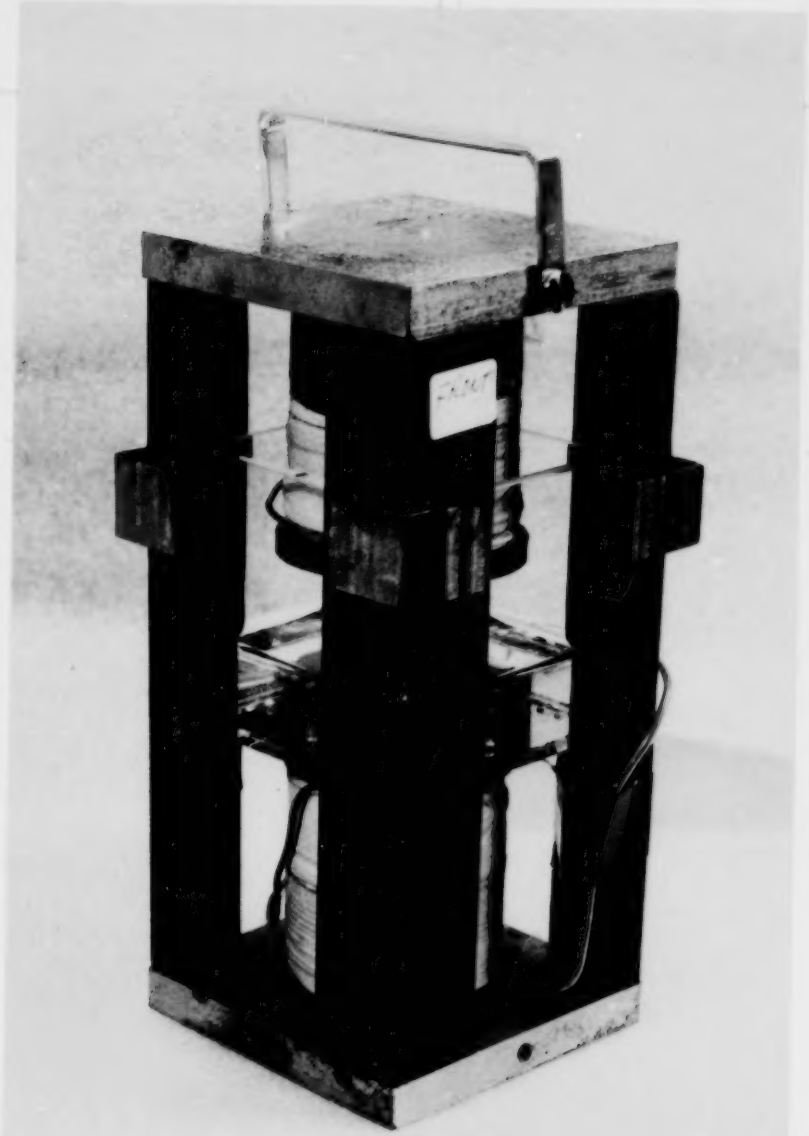

Figure 9. Magnet Assembly Used in the Modified Turbidimeter, Shown With Sample Cell in Position.

background value, equivalent to 0.023 NTU, occurred as a result of the modifications. The performance of the magnet is shown in Figure 10, and at a current of 20 amps the flux density in the gap of the magnet was 0.3 Tesla.

Using the modified turbidimeter, it was possible to measure the turbidity reading with the magnetic field off, and then to observe the change with the magnetic field on. A change in turbidity reading would be expected, the reasons for which are illustrated in Figure 11. With zero magnetic field, the fibers are in random orientations and they simply contribute to the total turbidity value. When the magnetic field is turned on, the turbidity reading will increase if the fibers align parallel to the field, because they present more light scattering area to the illuminating beam than if they are randomly oriented. If the fibers align in directions perpendicular to the field direction, they present less light scattering area to the light beam than they do if randomly oriented, and the turbidity reading will decrease. The change of reading will be specific to those species of fibrous particles which adopt preferred orientations in a magnetic field.

Performance of Modified Turbidimeter

The modified turbidimeter was calibrated using dispersions derived from the reference dispersions described earlier. The turbidity was measured initially, and then the change of reading was observed when the maximum value of magnetic field was applied.

Figure 12 shows the results obtained for UICC crocidolite, using the modified turbidimeter. Six separate experiments were made, and the results were found to be very reproducible. Unlike the situation in which actual turbidities were measured (described earlier), these measurements are not dependent on stray light variability or changes in the cleanliness of the sample cell between readings. The limitation appeared to be in the ability to read the output scale precisely. Figure 13 shows similar results for UICC amosite. The results for chrysotile are shown in Figure 14, and it is clear that chrysotile yielded a much lower change of turbidity than was the case for either crocidolite or amosite. Figure 15 shows the results for all three varieties of asbestos in terms of mass concentration. It should be emphasized that the points corresponding to the lowest concentrations in Figures 12-15 are not fundamental detection limits; the actual detection limits could be significantly improved by use of a more sophisticated difference signal measurement technique. Long term signal averaging would also allow further improvements in detection level.

The response of the modified turbidimeter system to other fiber and particle types was also examined, but without obtaining fiber counting information. These results are summarized in Table 4, and it can be seen that, as predicted, orientation of the n-alignment fiber types in the magnetic field results in a reduction of the turbidity reading. The results of Table 4 can be interpreted in a relative sense. For example, for the same turbidity, the increase produced by the magnetic field would be less for UICC amosite than for UICC crocidolite. This is probably because amosite contains

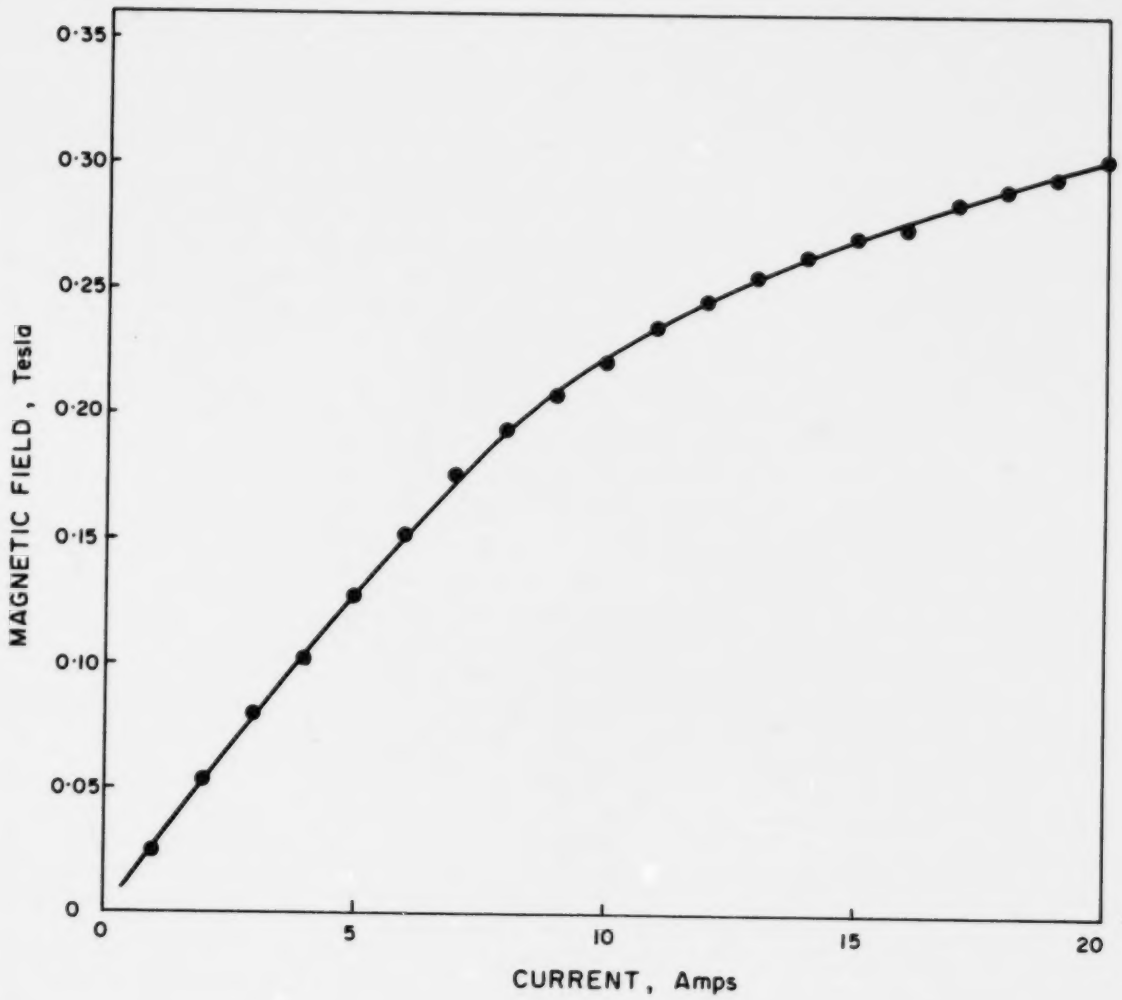

Figure 10. Calibration Curve for Magnet Assembly.

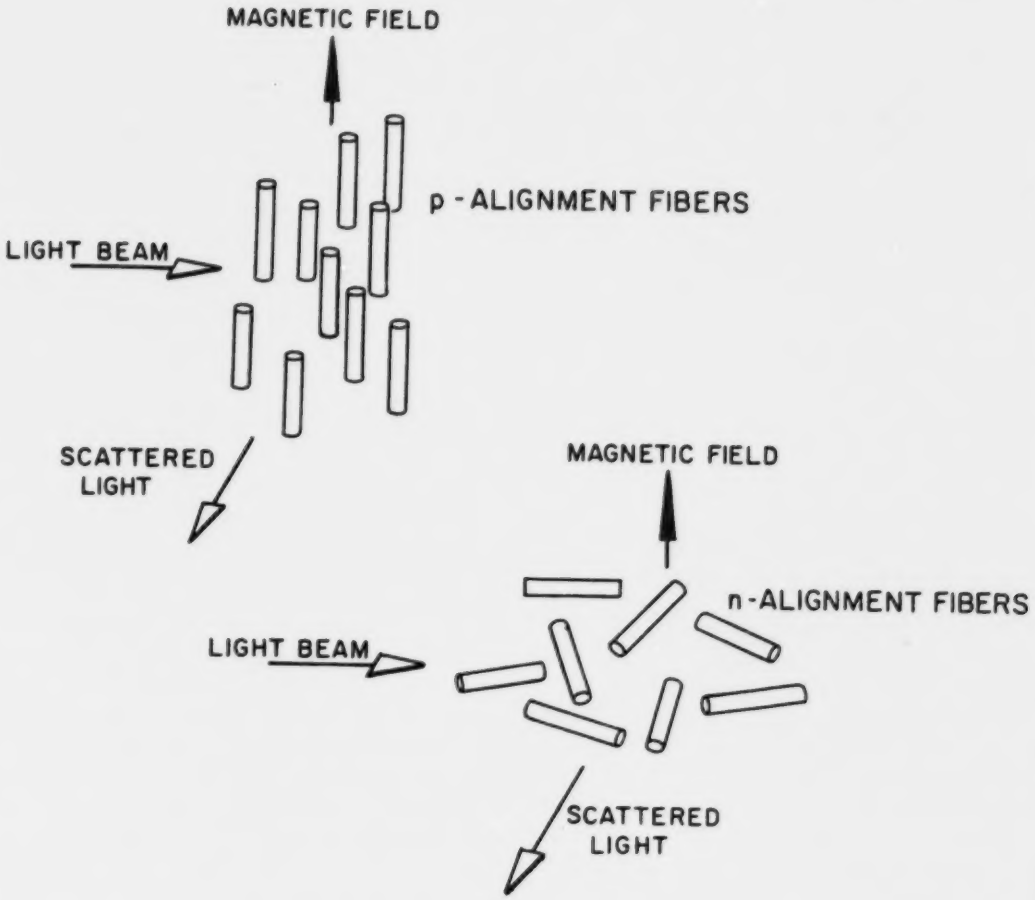

Figure 11. Illustration of Light Scattering by p- and n- Alignment Fibers.

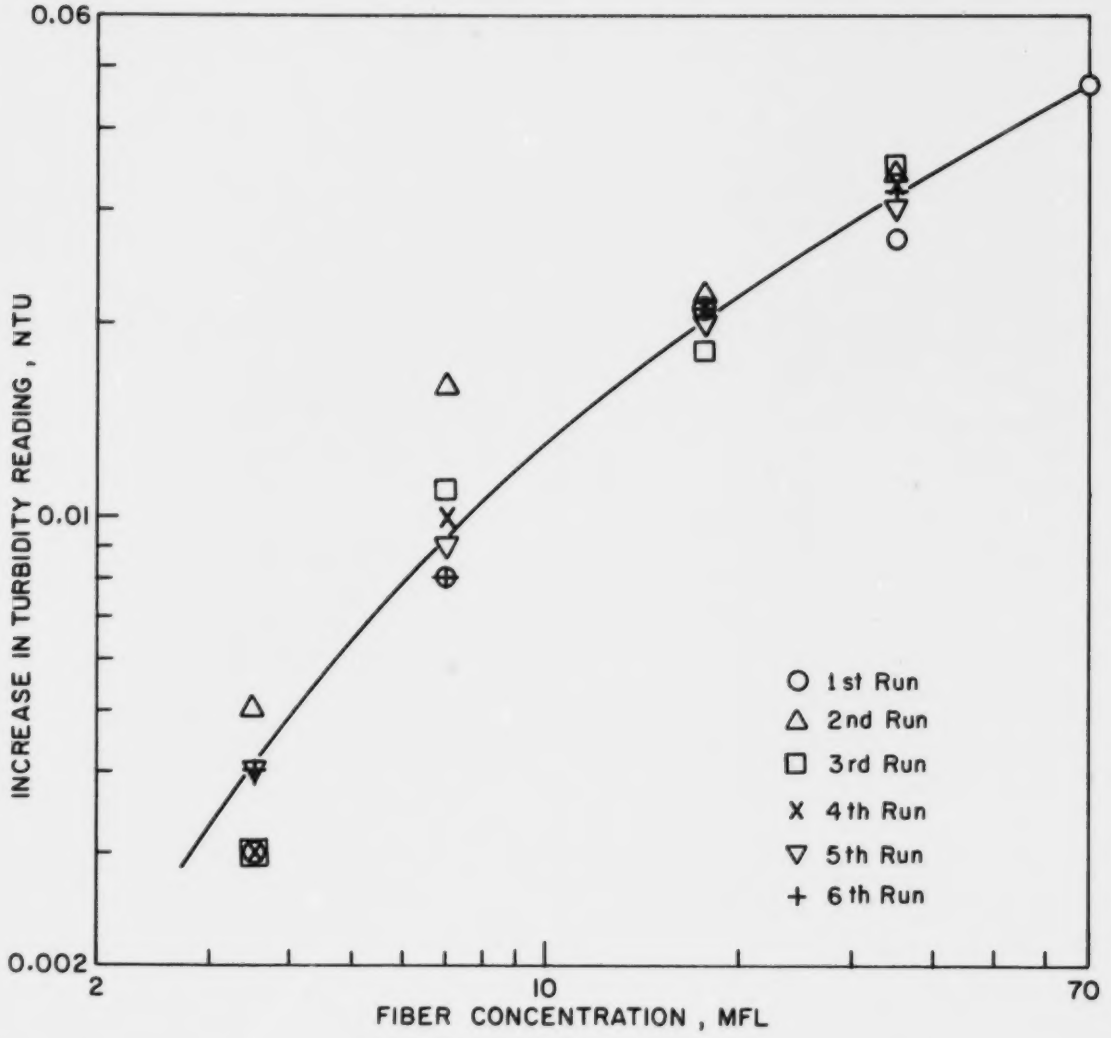

Figure 12. Effect of Magnetic Field on Turbidity Reading for UICC Crocidolite.

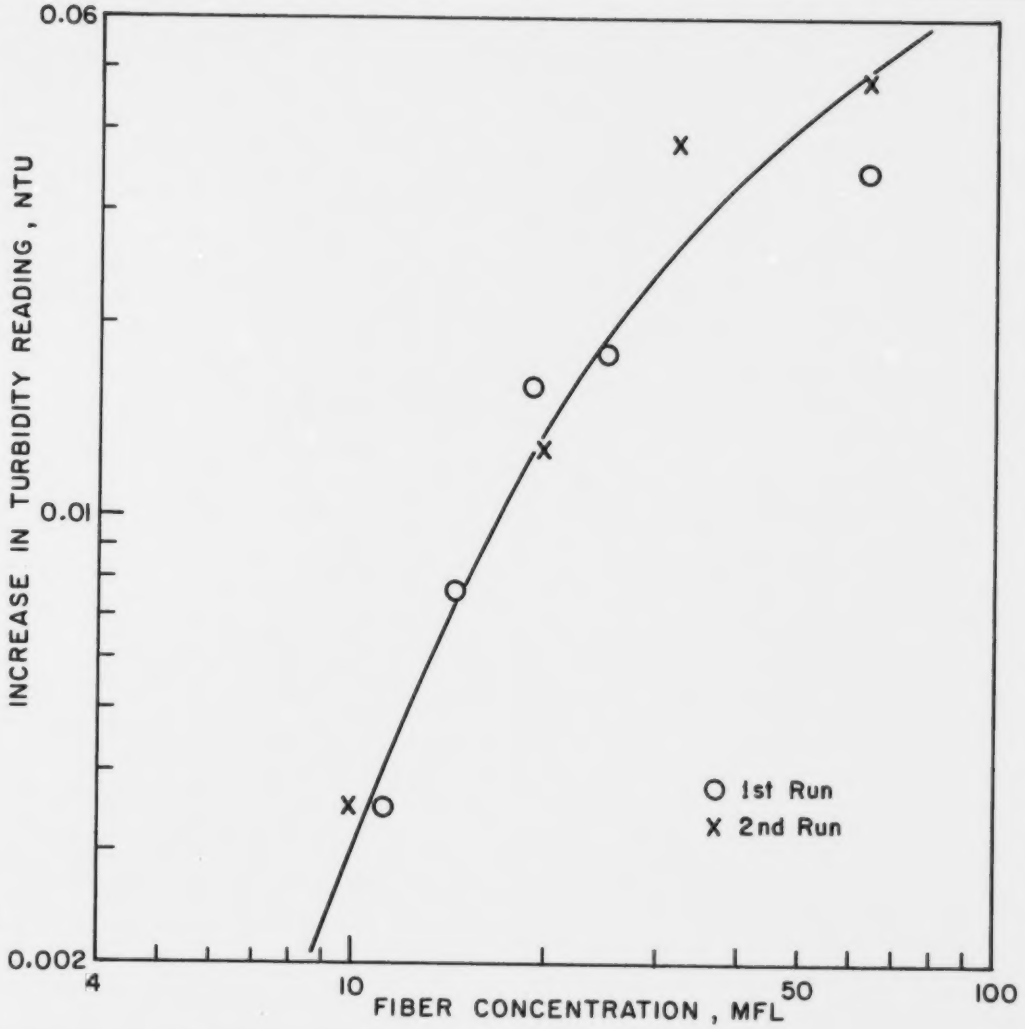

Figure 13. Effect of Magnetic Field on Turbidity Reading for UICC Amosite.

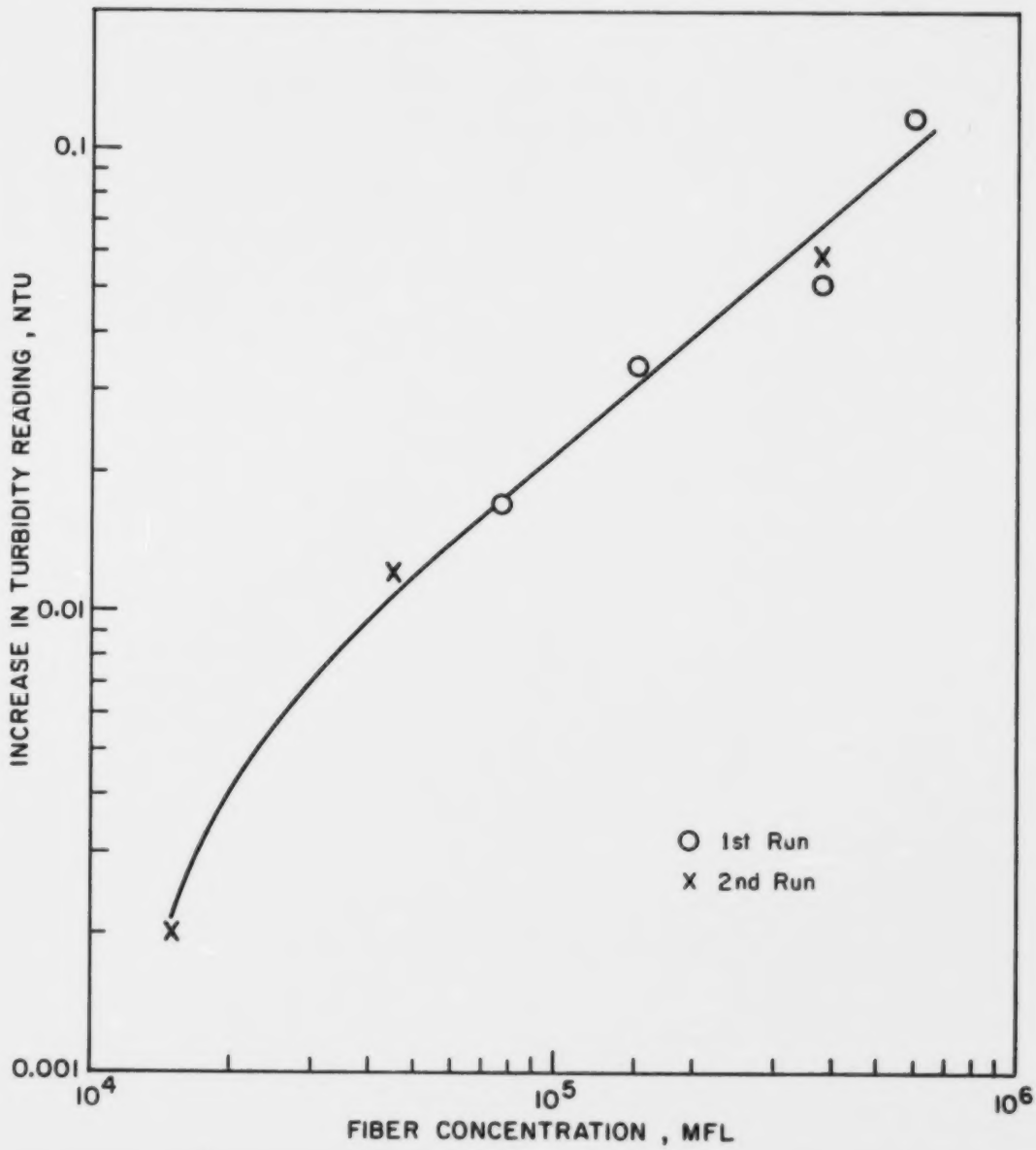

Figure 14. Effect of Magnetic Field on Turbidity Reading for Union Carbide Chrysotile.

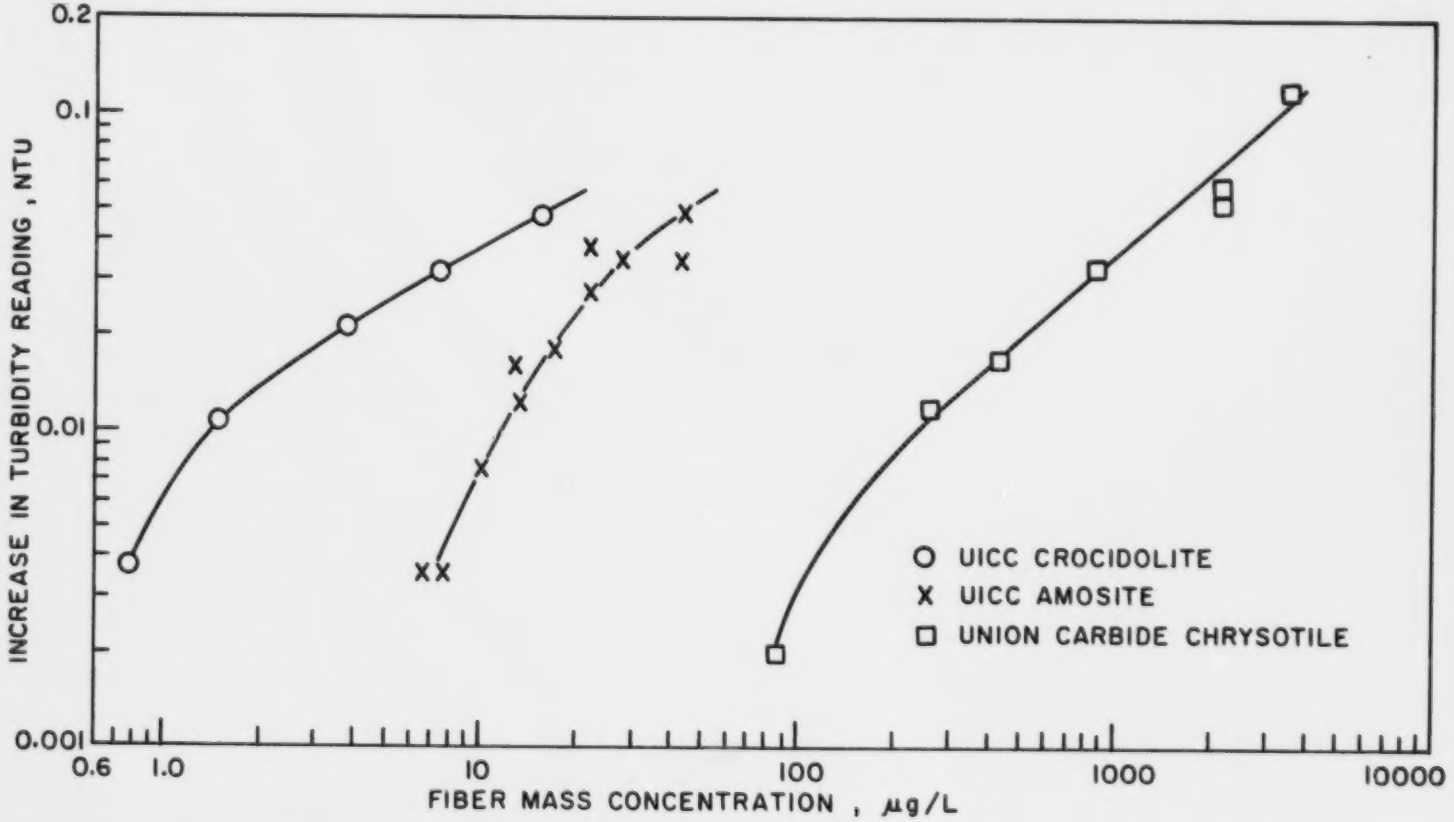

Figure 15. Effect of Magnetic Field on Turbidity Reading as Function of Mass Concentration for UICC Crocidolite, UICC Amosite and Union Carbide Chrysotile.

both p- and n-alignment fibers, and the effect of fiber alignment on the turbidity reading is partially self-cancelling. Theoretically therefore, there could be mixtures of fiber types for which the magnetic field would produce no change of turbidity reading. The behavior of a second variety of amosite (Lyndberg, R.S.A.) was examined, and the results are shown in Table 4. The response for the same value of turbidity was significantly higher than that for UICC amosite, probably indicating that there was a greater proportion of p-alignment fibers than in the case of the UICC material.

TABLE 4. EFFECT OF MAGNETIC FIELD ON TURBIDITY READINGS FOR SOME FIBROUS AND NON-FIBROUS PARTICLES

Material	Formazin Turbidity, NTU	
	No Magnetic Field	Change With 0.3T Magnetic Field
UICC Crocidolite	0.030	+0.047
UICC Amosite	0.030	+0.015
Amosite (Lyndberg, R.S.A.)	0.030	+0.040
Union Carbide Chrysotile	0.71	+0.051
UICC Anthophyllite	0.27	+0.012
Tremolite	1.50	-0.530
Actinolite	1.65	-0.370
Cummingtonite	0.89	+0.260
Ground Glass	1.20	0
Mississauga Tap Water	0.19	0

COMPARISON OF THE MODIFIED TURBIDIMETER WITH THE MAGNETIC ALIGNMENT - LIGHT SCATTERING SYSTEM

The magnetic alignment - light scattering system developed earlier by Chatfield and Riis[4] was shown to be capable of discriminating between the three commercial varieties of asbestos, on the basis of the shapes of the scattered light profiles. This is a major advantage of this system, compared with the modified turbidimeter which cannot discriminate between the fiber types. Figures 16-18 show the performance of the magnetic alignment - light scattering system, using the same fiber dispersions as those used to calibrate the modified turbidimeter. Examples of the

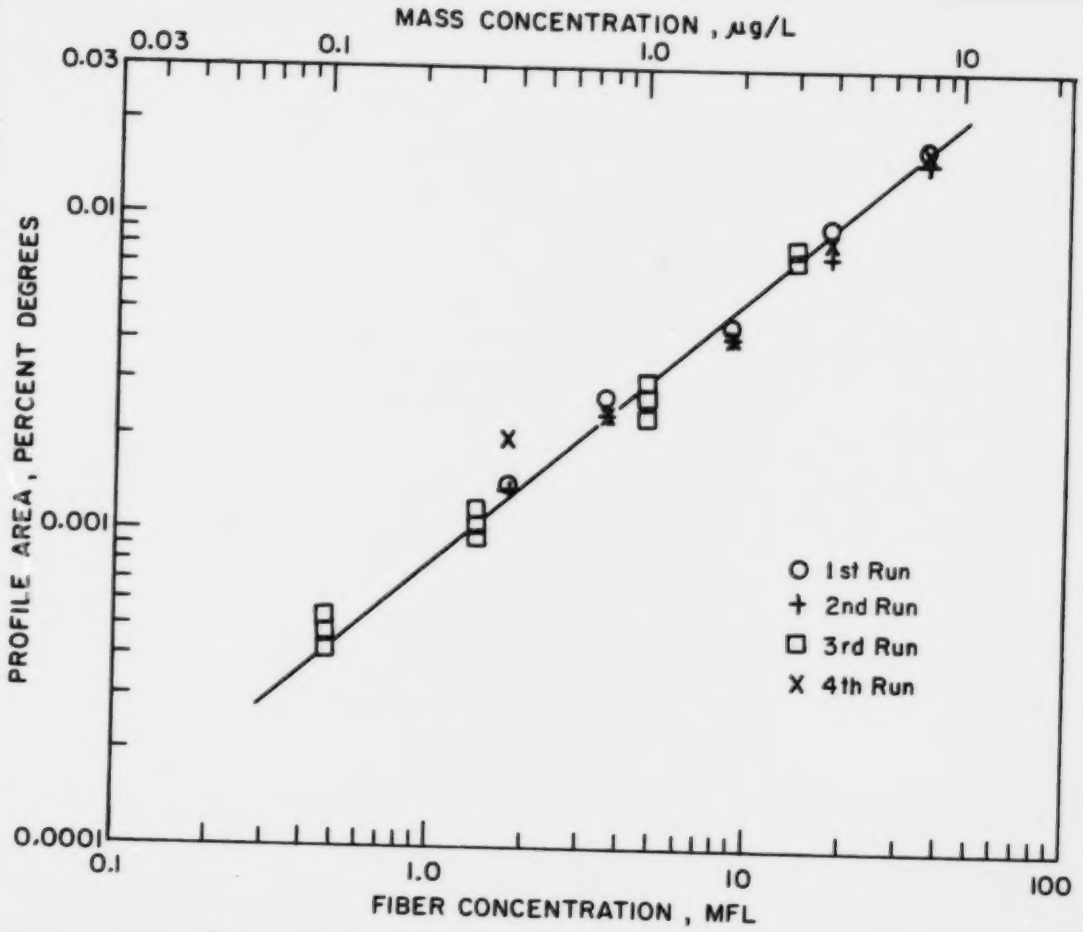

Figure 16. Performance of the Magnetic Alignment - Light Scattering System for UICC Crocidolite.

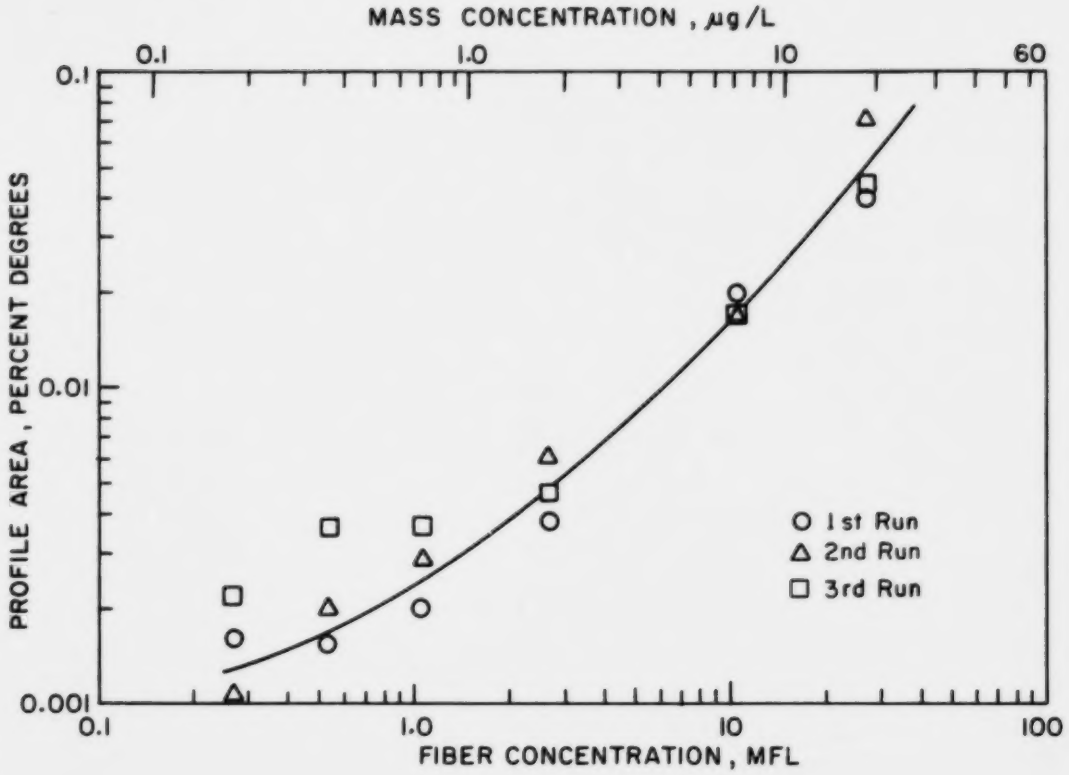

Figure 17. Performance of the Magnetic Alignment - Light Scattering System for UICC Amosite.

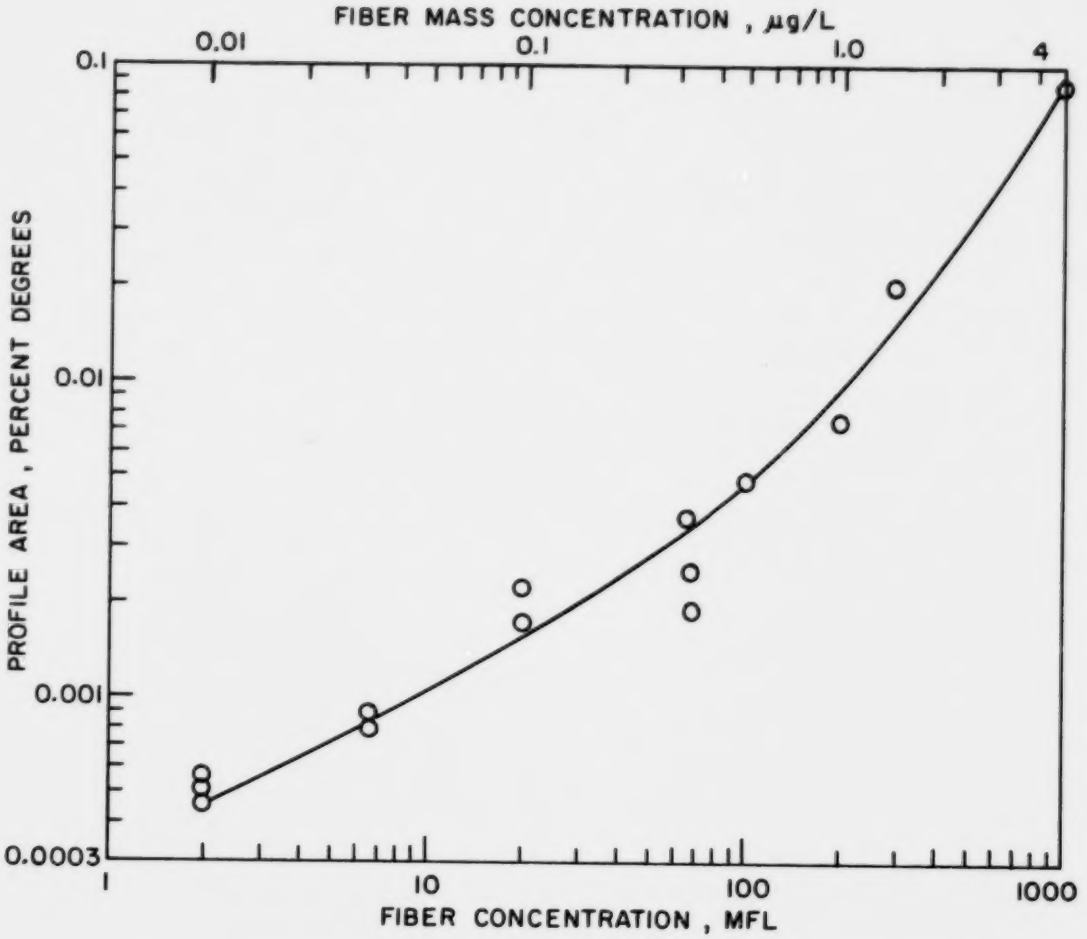

Figure 18. Performance of the Magnetic Alignment - Light Scattering System for Union Carbide Chrysotile.

scattered light profiles, and the processing of these to yield quantitative data, are shown in Appendix B.

Table 5 shows a comparison of the results from the turbidity measurements with those obtained using the magnetic alignment - light scattering system. It is not always possible to quote precise detection limits, because in some cases the measurements were limited by the coarseness of the turbidity read-out system. This is because the turbidity of the suspension was often such that the 2 mg/L reference standard was required when the fiber concentration was adequate to yield a measurable change in turbidity with the application of the magnetic field. With this reference standard, the 100 scale divisions of the read-out drum correspond to increments of 0.007 NTU each rather than the value of 0.0017 NTU when using the 0.5 mg/L standard. A difference reading of 0.004 NTU was therefore difficult to obtain accurately. In deriving the detection levels in Table 5, it has been assumed that 0.004 NTU is a reasonable limit for reliable detection of turbidity exceeding that of a distilled water blank, and that a change of 0.004 NTU could be detected when the modified turbidimeter was in use. The detection levels for the magnetic alignment system, and those for the modified turbidimeter, are capable of some improvement by application of more sophisticated data read-out methods.

SAMPLE PREPARATION

Fiber Concentration Requirements

The detection limit of the Sigrist L65 instrument in practice corresponds to a turbidity of about 0.002 NTU. It is unlikely, however, that many drinking water samples could be processed to yield turbidities less than twice this, or 0.004 NTU. The detection levels of asbestos fibers quoted in Table 5 represent the fiber concentrations to which this value of turbidity corresponds if only asbestos is present. However, an asbestos fiber concentration reported on the basis of a single turbidity measurement can be regarded only as the maximum fiber concentration consistent with that turbidity measurement, the actual fiber concentration may be much lower and the balance of the turbidity value would be a result of other suspended particulate. The only way in which this simple measurement technique can be made more sensitive to low levels of asbestos would be to concentrate the asbestos fibers specifically and to reduce the turbidity of the concentrated dispersion to a value close to the instrumental detection level. For example, in order to achieve a detection level of 1 MFL of chrysotile, it would be necessary to concentrate the chrysotile by a factor of 1000, and at the same time reduce the turbidity of the concentrated dispersion to a value no higher than 0.004 NTU. Figure 19 shows the possible methods by which fiber concentration could be achieved. The procedures are based on ozone-ultraviolet light (UV) treatment (to destroy the organic materials), magnetic separation, fiber-selective filtration and also the fiber scavenging effect reported by Chatfield et al[13].

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TABLE 5. COMPARISON OF RESULTS FROM TURBIDITY MEASUREMENTS AND MAGNETIC ALIGNMENT - LIGHT SCATTERING

Technique	Approximate Detection Level MFL (μg/L)			Comments
	UICC Crocidolite	UICC Amosite	Union Carbide Chrysotile	
Turbidimeter	5 (1.3)	2 (1.3)	1000 (6)	For a turbidity measurement of 0.004 NTU
Modified Turbidimeter* (Fiber-Specific Readings)	3.5 (0.8)	12 (7.5)	20000 (100)	Results specified for a change of 0.004 NTU
Magnetic Alignment - Light Scattering	0.5 (0.1)	0.5 (0.3)	2 (0.01)	

*These results are limited by the inability to read smaller difference signals on the turbidimeter. Use of time averaging and digital output techniques would yield significantly lower detection levels.

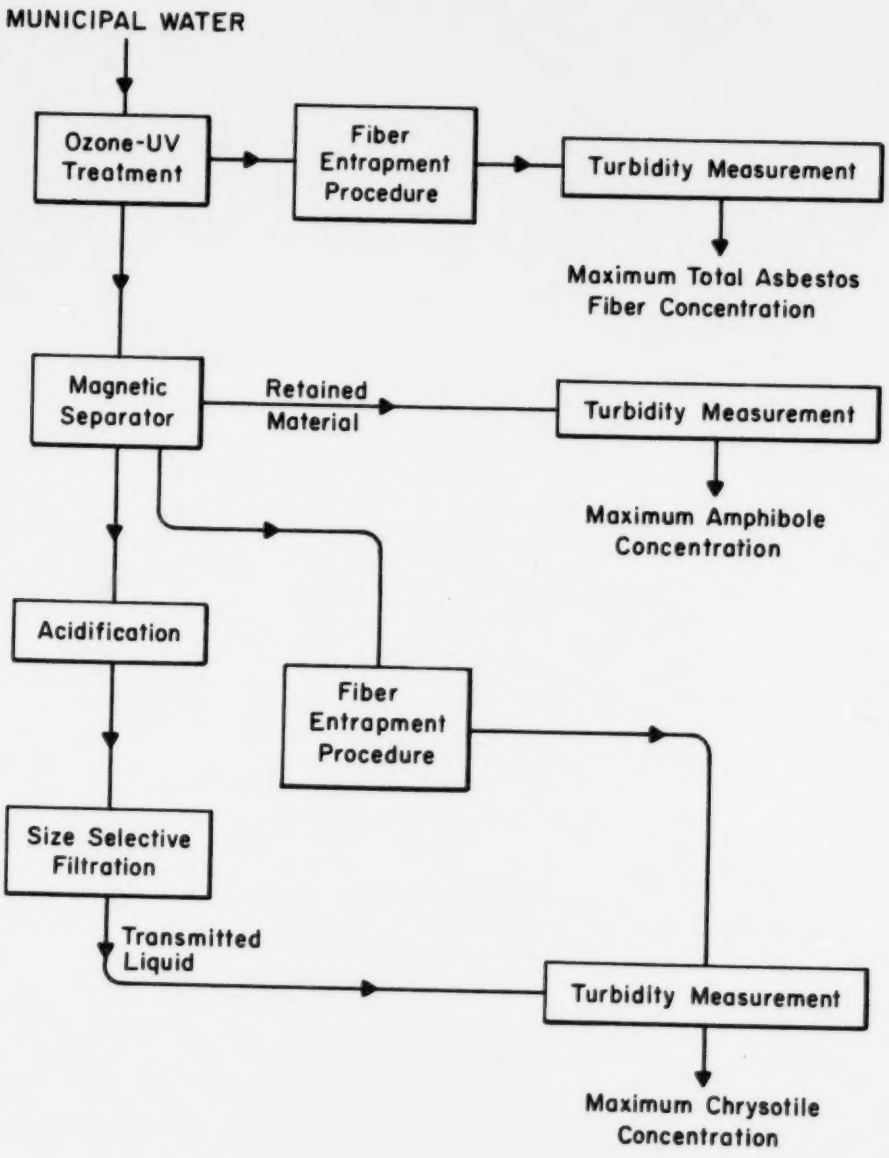

Figure 19. Possible Techniques for Selective Fiber Concentration Prior to a Turbidity Measurement.

Turbidity Reduction and Selective Fiber Concentration

Much of the suspended material which contributes to turbidity in drinking water is organic. There is also evidence that many of the chrysotile fibers in drinking water sources become attached to organic materials,[13] and if selective fiber concentration techniques are to be successful the fibers must be released from attachment to other materials. Organic materials can be oxidized using ozone-UV treatment.[3] This treatment reduces the turbidity of the sample, reduces the aggregation problems and allows the oxidation to be accomplished with minimum disturbances to the sample.

Techniques for selective fiber concentration depend on the variety of fiber present. The magnetic amphiboles, i.e. those with a substantial iron concentration such as crocidolite or amosite, can be very effectively concentrated by magnetic separation. Early work was reported by Timbrell[20], and later by Chatfield and Riis[4]. However, although in both of these studies the collection characteristics of simple magnetic separators were studied, no work was reported on recovery of the fibers from the magnetic separator.

Three experiments were carried out to investigate magnetic separation and recovery of UICC crocidolite from Mississauga tap water. The test samples were each prepared by mixing one reference ampoule of UICC crocidolite with 4 liters of Mississauga tap water. The concentration of the crocidolite in these test samples was about 2.8 MFL. The apparatus used was similar to that reported by Timbrell, and by Chatfield and Riis. A loosely-compacted ball of type 430 stainless steel wool (Thamesville Metal Products Ltd, Thamesville, Ontario, Canada) weighing 1 gram was placed at the bottom of a buret, and positioned between the poles of an electromagnet in a field of 1.3 T. Each test sample was passed through a separator at a flowrate of 8 mL/minute. After the liquid had all passed through, the buret was removed from the magnetic field. The end of the buret was submerged in 40 mL of non-boiling still water in a 50 mL disposable plastic beaker in an ultrasonic bath. The water in the beaker was allowed to rise through the steel wool in the buret several times during this treatment, and then 10 mL of water was drained through the buret as a final rinse and collected with the water in the disposable beaker. The water in the beaker was then analyzed for asbestos by the TEM analytical method[3]. The results are shown in Table 6. It is clear from these results that there was some difficulty in either the separation or the recovery of the smaller fibers, which is indicated by the variability of the numerical recovery. The low recoveries were possibly a consequence of the high flowrate through the separator. In these experiments a flowrate of 8 mL/min was used, compared with the 2 mL/min used by Timbrell[20] and Chatfield and Riis[4]. The higher flowrate was necessary to provide more realistic filtration times. The fact that the mass recovery was nearly constant at about 65% indicates that the larger fibers, representing most of the mass, were separated and recovered in a reproducible manner. Although some improvement in the technique would be desirable, the concentration technique was very simple and effective. In

summary, a fiber concentration of 2.8 MFL in a 4 liter volume of water was concentrated in three separate experiments to 31.7 MFL, 40.2 MFL and 98.1 MFL in a volume of 50 mL, thus yielding concentration factors between 11.3 and 35.0. In terms of mass, a concentration of 0.63 µg/L was concentrated from 4 liters of water to yield a concentration of about 33 µg/L in a 50 mL volume. This represents a mass concentration factor of 52.

TABLE 6: RECOVERY OF UICC CROCIDOLITE FROM MAGNETIC SEPARATION

SAMPLE	NUMERICAL RECOVERY*,%	MASS RECOVERY*,%
1	43	64
2	18	67
3	14	65

*Results based on counts of approximately 200 fibers.

Magnetic separation cannot be used to concentrate fibers which do not contain a high concentration of iron. Chrysotile and amphiboles such as tremolite and anthophyllite fall into this category. The size distribution of waterborne chrysotile in drinking water supplies usually has a median fiber length between 1.0 and 2.0 µm, and it was thought that a selective filtration technique could possibly achieve some concentration of the fibers relative to larger suspended particles of other materials. The collection efficiency curves of Nuclepore polycarbonate filters for chrysotile fibers were therefore required in order to select an appropriate pore diameter for this procedure. These curves were determined and the results are shown in Figure 20. The technique by which these measurements were made was developed by Couchman and Moseley[21], and the full procedure is described in Appendix C. It can be seen in Figure 20 that even the 0.8 µm pore diameter Nuclepore filter has a collection efficiency of less than 40% at a fiber length of 2 µm. If it were demonstrated that the turbidity could be significantly reduced by filtration through such a filter, and at the same time most of the chrysotile fibers passed through, the technique would offer a simple selective fiber concentration method.

The results of the turbidity reduction experiments are shown in Tables 7 and 8. The water samples were first treated using the ozone-UV technique, and aliquots of the product were then filtered through unbacked 0.8 µm, 2.0 µm and 3.0 µm pore diameter Nuclepore filters. The oxidation of organic materials was carried out first, because it has been demonstrated

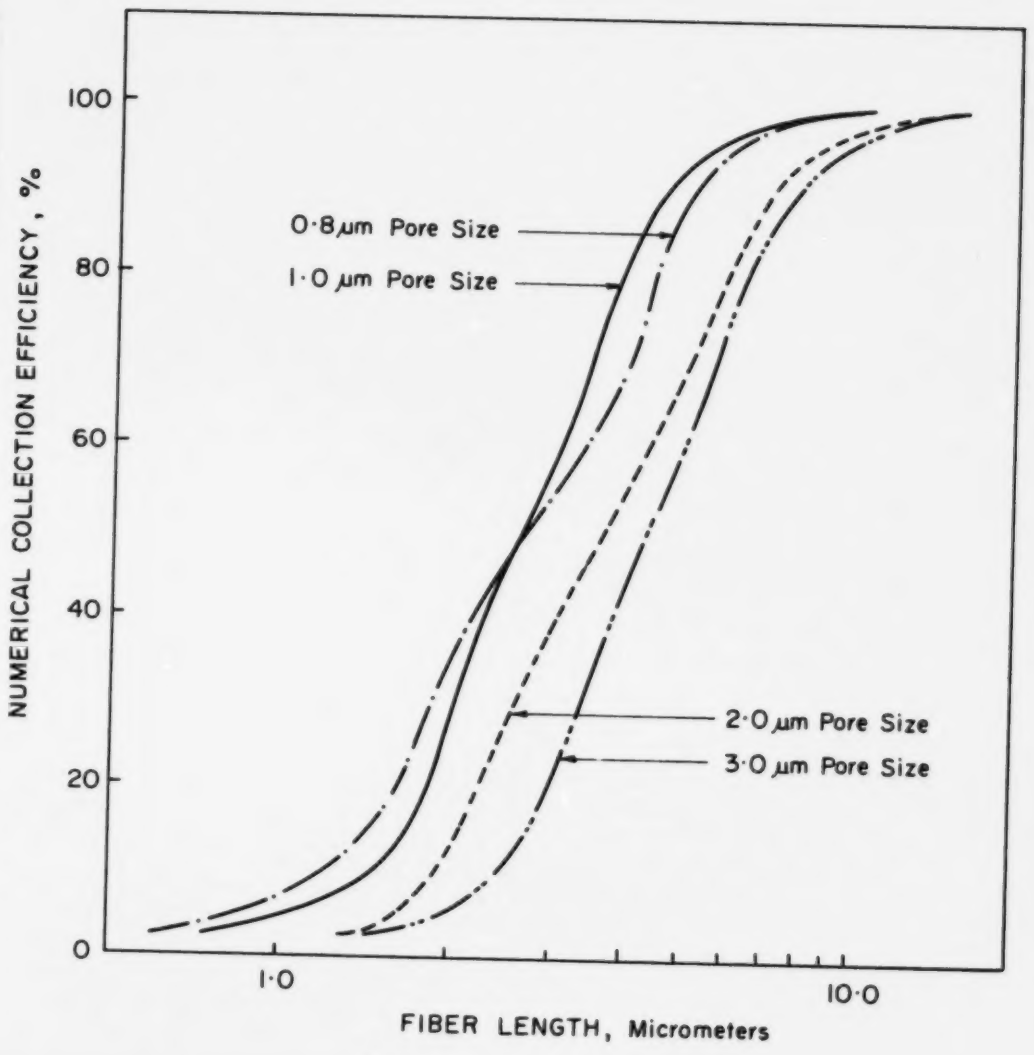

Figure 20. Numerical Collection Efficiency for Single Fibril Chrysotile as a Function of Fiber Length Using 0.8 µm, 1.0 µm, 2.0 µm and 3.0 µm Pore Size Nuclepore Filters (based on counts of approximately 300 fibers).

TABLE 7: RESULTS OF TURBIDITY REDUCTION EXPERIMENTS ON MISSISSAUGA TAP WATER

Treatment	Turbidity, NTU	
	Sample 1	Sample 2
1. Background Measurement using non-boiling still water	0.0017	<0.0017
2a. Mississauga Tap Water	0.109	0.109
2b.* Mississauga Tap Water Filtered through a Nuclepore Filter of:		
0.8 µm Pore Diameter	0.069	0.065
2.0 µm Pore Diameter	0.103	0.102
3.0 µm Pore Diameter	0.107	0.105
3a. Mississauga Tap Water after 1 hour of ozone-UV treatment	0.027	0.028
3b.+ After a further 3 hours of ozone-UV treatment	0.054	0.056
3c. Filtration of the product from 3b through a Nuclepore Filter of:		
0.8 µm Pore Diameter	0.0058	0.0066
2.0 µm Pore Diameter	0.0165	0.0165
3.0 µm Pore Diameter	0.023	0.026

*Without the use of ozone-UV treatment prior to filtration, any asbestos fibers may be attached to either the container or to other particulate material retained by the filter.

+Increase in turbidity with further ozone-UV treatment is a consequence of oxidation products insoluble in water. Such products include calcium oxalate, which can be dissolved by addition of acid immediately prior to filtration.

that these materials become attached to chrysotile asbestos fibers, and such aggregates of material are more likely to be collected by the Nuclepore filter during the selective filtration step. The results for duplicate samples of Mississauga tap water shown in Table 7 indicate that the combination of the ozone-UV treatment and filtration was highly effective in reducing the turbidity. When a 0.8 μm pore diameter filter was used, the turbidity was reduced by factors of about 19 and 17 for the duplicate samples. Using 2.0 μm pore diameter filters, the corresponding turbidity reduction factor was 6.6 for both samples. Table 8 shows corresponding results for Sherbrooke tap water and Duluth raw water. For Sherbrooke tap water, the procedure yielded results very similar to those obtained with Mississauga tap water. The results for Duluth raw water indicate that the technique was less effective, but nevertheless, turbidity reduction factors of 8.8 and 3.4 were achieved for 0.8 μm and 2.0 μm pore diameter filters respectively.

TABLE 8: RESULTS OF TURBIDITY REDUCTION EXPERIMENTS ON SHERBROOKE TAP WATER AND DULUTH RAW WATER

Treatment	Turbidity, NTU	
	Sherbrooke Tap Water	Duluth Raw Water
1. Background Measurement using non-boiling still water	0.0020	0.0017
2. Untreated Sample	0.078	0.333
3.* Filtration through Nuclepore Filter of:		
0.8 μm Pore Diameter	0.019	0.030
2.0 μm Pore Diameter	0.030	0.054
3.0 μm Pore Diameter	0.037	0.058
4. Sample after 3½ hours of ozone-UV treatment	0.053	0.311
5. Filtration of the product of 4 through a Nuclepore Filter of:		
0.8 μm Pore Diameter	0.005	0.038
2.0 μm Pore Diameter	0.016	0.099
3.0 μm Pore Diameter	0.019	0.124

*Without the use of ozone-UV treatment prior to filtration, any asbestos fibers may be attached to either the container or to other particulate material retained by the filter.

Although a useful reduction in the turbidity of a water sample can be achieved by these procedures, this would be of no value if there were also a proportional collection of chrysotile fibers by the filter. The collection efficiency curves indicate that much of the chrysotile should pass through the filters, but this is not necessarily the case if it is dispersed with other particulate in a drinking water sample. Experiments were performed using samples prepared by mixing Mississauga tap water with a reference chrysotile dispersion. A 5 mL control sample of the reference dispersion was analyzed, and the test samples were prepared by adding 45 mL of the reference dispersion to 100 mL of Mississauga tap water. Each of these test samples was filtered through an unbacked Nuclepore filter of either 1.0, 2.0 or 3.0 µm pore diameter, and the filtrate was then analyzed. The vacuum was maintained at 17 kPa during the filtration. The samples for analysis were filtered through 0.05 µm pore diameter Nuclepore filters, and TEM specimens were prepared from these filters. An additional experiment was performed using a 2.0 µm pore diameter Nuclepore filter, in which the test sample was treated using the ozone-UV method before the test. The results are shown in Table 9. It can be seen, as would be expected, that there was a progressive increase in the transmission of fibers through the filter as larger pore diameter filters were used. The experiment in which the sample was oxidized, prior to filtration through a 2.0 µm pore diameter filter, shows that about 78% of the fibers passed through into the filtrate. In terms of mass concentration, 73% was recovered in the filtrate relative to the mass concentration of the control sample. The results confirm that the oxidation step should be carried out prior to the selective filtration. Although the experiment incorporating oxidation and a 1.0 µm pore diameter filter was not performed, it is likely that this would yield a recovery of about 50%.

TABLE 9. RESULTS OF EXPERIMENTS TO MEASURE THE TRANSMISSION OF TEST CHRYSOTILE DISPERSIONS THROUGH NUCLEPORE FILTERS

Filter Pore Diameter µm	Concentration in Filtrate*, MFL		Estimated Mass Concentration, µg/L
	Mean	95% Confidence Interval	
1.0	75.3	62.1 - 88.4	0.20
2.0	91.6	75.6 - 108	0.38
2.0**	155	129 - 181	0.65
3.0	105	84.7 - 126	1.04
Control Sample	200	174 - 227	0.89

*Concentration refers to equivalent concentration in original reference standard.

**Sample oxidized with ozone-UV treatment prior to filtration.

Another method which can potentially be used for selective concentration of chrysotile was reported by Chatfield et al[13], who observed that under some circumstances the inside surfaces of plastic containers appeared to scavenge chrysotile fibers from suspension. It was demonstrated that the scavenged fibers would be released from the surfaces by carrying out an ozone-UV oxidation conducted inside the sample container. In experiments using artificial suspensions of chrysotile in double-distilled water, the fibers were found to be completely collected by the container walls after shaking on a laboratory reciprocal shaker for only 24 hours. In experiments using tap water from Sherbrooke, Quebec, (a supply known to contain chrysotile fibers) it was shown that, after periods of storage in polyethylene bottles exceeding 40 days, between 66% and 86% of the chrysotile fibers in the sample had been collected by the inside surfaces of the bottles. Experiments using Mississauga tap water, with addition of an artificial dispersion of chrysotile, showed a similar effect, although the magnitude was somewhat variable. It was concluded that the effect was probably due to the presence of polysaccharides, produced by biological activity in the sample. Bacteria capable of producing these materials are often present in drinking water, and it has been shown that polysaccharides are strongly adsorbed by chrysotile as a consequence of its very high positive zeta potential.[22] More recently, Lauth and Schurr[23] have demonstrated that chrysotile exhibits an affinity for a variety of algae, cryptomonas erosa, and suggest that this may also occur with other varieties.

Experiments were carried out to determine if the fiber scavenging effect could be enhanced by increasing the surface area of polyethylene inside the container. Dispersions of Union Carbide chrysotile fibers were prepared in double-distilled water from the same source as that used in the earlier work. One set of polyethylene bottles was filled with polyethylene coils, thus increasing the total area of polyethylene surface from the 560 cm^2 of the bottle alone to a total of 1260 cm^2. About 800 mL of the fiber dispersion were placed in each bottle, and the bottles were then shaken for periods of 8, 16 and 24 hours. After the shaking period was completed, a sample of the water from each bottle was filtered for TEM analysis. The bottle was then emptied, and re-filled with fresh double-distilled water. The inside of the bottle was treated using the ozone-UV technique, and a sample of the water was filtered for TEM analysis. Two similar experiments were also carried out using Mississauga tap water. The results are shown in Table 10, in which it can be seen that in no case did the separation efficiency of the technique exceed 20%. This is lower than the values reported in the earlier work, and is not adequate for use in an analytical procedure. The reasons for the low values of recovery have not been resolved, but may possibly be that the organisms present in these samples were different, or that there were too few of them to be effective. It is also possible that the fiber capture mechanism operates in two stages: (a) the fibers adhere on contact with an organism; and, (b) the organic materials subsequently aggregate or become attached to the container surfaces. A review of past experiments already reported[13] indicates that almost complete removal of chrysotile fibers from suspension can be readily achieved. However, in most of these past experiments, the primary interest

TABLE 10. RESULTS OF FIBER SEPARATION EXPERIMENTS

Sample	Shaking Time Hours	Separation Efficiency, %
Double-Distilled Water No Additional Polyethylene Inside Container Internal Surface Area: 560 cm^2	8	0
	16	10
	24	20
Double-Distilled Water Polyethylene Coils Inside Container Total Surface Area: 1260 cm^2	8	20
	16	15
	24	13
Mississauga Tap Water No Additional Polyethylene Inside Container Internal Surface Area: 560 cm^2	24	16
	24	16

was to remove such interferences from the analytical method; accordingly, the sample was treated using the ozone-UV procedure without attempting any separation. Therefore, there is an ambiguity of interpretation as to whether the fibers became attached to the container, or whether they were collected by particles of the organic material which then simply coagulated to form a few large aggregates. In either case, the fiber count would have been reduced, either because the fibers were no longer in suspension or because the fibers were associated in a small volume of coagulum from which a representative sample could not be obtained. Nevertheless, in several experiments, very high separation efficiencies were obtained, and the procedure could become a very effective separation technique if the mechanism could be elucidated and brought under control. This was not considered to be possible within the constraints of this program, and no further experiments were carried out on this procedure.

SECTION 6

DISCUSSION

The Sigrist L65 turbidimeter was selected as the instrument which most closely met the needs of this investigation. The null principle of the instrument and the use of permanent turbidity reference standards allows lower detection limits to be obtained than are possible with any other instrument examined. The actual reading for non-boiling still water was constant to within half a scale division on the highest sensitivity scale, if the same sample cell was used in the same orientation and it was thoroughly cleaned before use. Under these conditions, the reading obtained corresponded to 0.09 mg/L SiO_2. This reading is equivalent to about 0.0297 NTU, which represents the combined effects of instrumental stray light and scattering by the non-boiling still water. The effect of the stray light alone can be measured using the procedure defined by the manufacturer, and it was determined to be equivalent to 0.04 mg/L SiO_2. The actual turbidity of the non-boiling still water was therefore equivalent to 0.05 mg/L SiO_2, or about 0.017 NTU. When attempting to calibrate the system against formazin standards, a difficulty arises at low values of turbidity, since the calibration is based on successive dilution of a 40 NTU standard with "turbidity-free water". In this investigation, the calibration against formazin has been made assuming the dilution water to have zero turbidity. Accordingly, at low values of turbidity, the values quoted should not be interpreted as absolute turbidity, but rather as a contribution by the material deliberately added to the dispersion being measured.

The relationships obtained between turbidity and asbestos fiber concentrations indicate that for water with a turbidity of only 0.1 NTU, asbestos fibers at realistic concentrations do not contribute significantly to the total turbidity. A contribution of only 0.01 NTU, for example, corresponds to 17 MFL, 7 MFL and 3500 MFL for crocidolite, amosite and chrysotile respectively. Many treated water supplies have turbidities significantly in excess of 0.1 NTU, and if asbestos is present at the 1 MFL level, its contribution to the total turbidity signal will be insignificant.

In order for a measurement of turbidity to be meaningful as a direct indicator of asbestos fiber concentration, it would be necessary to reduce the turbidity of the sample such that the asbestos contributes a significant proportion of the scattered light signal. Then it must be recognized that this value represents only the maximum fiber concentration which may be in the sample, since other types of particle could be responsible for turbidity measurement. If it were possible to reduce the turbidity of the water sample to 0.004 NTU, the detection levels were shown to correspond to

5 MFL, 2 MFL and 1000 MFL for crocidolite, amosite and chrysotile respectively. In order to achieve a detection limit of 1 MFL, therefore, the fibers in the sample would require concentration by corresponding factors of 5, 2 and 1000, in addition to the requirement that the turbidity of the concentrate be reduced to a value no higher than 0.004 NTU.

The response of the turbidimeter varied with the different species of fiber used in this investigation. While minor variations in response might be expected, the results show that chrysotile produced less turbidity than the same mass concentration of either crocidolite or amosite. It is most likely that the variation was a consequence of the large difference between the fiber diameter distribution of the chrysotile and those for the crocidolite and amosite. This observation shows that turbidity measurements could not be interpreted in terms of fiber concentrations without a knowledge of the fiber size distribution, and possibly the species of fiber.

The modified turbidimeter gave readings which were specific to the fiber concentrations of the samples. Two readings of turbidity were made for each measurement, one with zero magnetic field, and one with a magnetic field of 0.3 T applied to the sample. A difference of 0.004 NTU between the two readings was detectable with the present configuration of the modified instrument, which leads to detection limits of 3.5 MFL, 12 MFL and 20000 MFL for crocidolite, amosite and chrysotile respectively. These are not the fundamental detection limits for the modified turbidimeter, since the measurement appeared to be primarily limited by the ability to read a small difference on the scale. The use of digital techniques for measurement of the off-balance signal, and time averaging of the difference signal, would certainly allow a significant reduction of these detection limits.

It was found that fibers which aligned parallel to the magnetic field direction caused an increase in the turbidity signal, and those which adopted orientations normal to the magnetic field direction caused a decrease in the turbidity signal. This was in accordance with theory. The modified turbidimeter was therefore capable of discrimination between p- and n-alignment fibers, although in materials where both are present, the opposing effects on the turbidity cancel, possibly leading to an incorrect conclusion that no asbestos is present if the relative proportions are such that complete cancellation occurs. Partial cancellation of the signal occurred in the case of UICC amosite, and it was found that another variety of amosite (Lyndberg, R.S.A.) yielded a significantly greater change in turbidity. Clearly, the response of the modified instrument to amosite will depend critically on the relative proportions of p- and n-alignment fibers. No change in turbidity signal was observed for either Mississauga tap water or dispersions of ground glass, indicating that the modified instrument only responds to elongated particles.

The magnetic alignment - light scattering system of Chatfield and Riis[4] has concentration detection limits much lower than those of either version of the turbidimeter, and the system requires a sample volume of only 5 mL, rather than the 130 mL or 65 mL required for the turbidimeter. These detection limits were 0.5 MFL, 0.5 MFL and 2 MFL respectively for

crocidolite, amosite and chrysotile. The low detection limits are a consequence of the fact that the system incorporates time averaging and is also designed for measurement of small differences between scattered light signals. A major advantage of the system is that a profile is obtained which represents the scattered light intensity as a function of the angle between the magnetic field direction and the plane of the optic axis. This allows the effects of p- and n-alignment fibers to be measured separately, whereas in the modified turbidimeter these effects partially cancel.

From the routine monitoring point of view, it would be desirable if the turbidimeter method allowed detection of fiber concentrations down to about 1 MFL or lower. It would therefore be necessary to carry out some kind of concentration step prior to making the turbidity measurement. It is important that any concentration steps used should be simple. If these steps were to involve several hours of labor per sample, the method would not be useful, because the cost and time for analysis could then be matched by a simplified version of the TEM method incorporating only rudimentary fiber identification.

The amphiboles which contain significant proportions of iron can readily be separated from most of the other suspended particulate in a water sample by magnetic separation. These amphiboles include crocidolite, amosite and some of the actinolites. The experiments using crocidolite show that a useful pre-concentration can be achieved very simply. A 4 liter sample is first oxidized using the ozone-UV treatment. This eliminates most of the organic materials, and ensures that the fibers are not associated with such materials during the separation step. The sample is then passed through a simple magnetic separator, after which the material collected by the separator is re-dispersed in 65 mL of clean water. Although the concentration factor based on numbers of fibers for some reason varied between 8.7 and 26.9, the concentration factor for fiber mass was constant at about 40. Earlier work[4] showed that amosite could also be collected with high efficiency by this technique. Using the magnetic separation technique to concentrate the fibers from a 4 liter water sample, the detection limits of the turbidity measurements can be improved by a factor of 10 or more for the high-iron amphiboles. This would enable fiber levels lower than 1.0 MFL and 0.4 MFL to be detected for crocidolite and amosite respectively, using the turbidimeter in its original configuration. In the modified turbidimeter, the measurement would be specific to fibers, and the corresponding detection limits would be lower than 0.35 MFL and 1.2 MFL for crocidolite and amosite respectively. Since the turbidity measurement appears to be more closely associated with the relative mass of fibers than with the number of fibers, the relevant concentration factor in terms of turbidity measurement may be the concentration factor of about 40 for fiber mass. The magnetic separation efficiency for a particular fiber species is probably a function of fiber size distribution, and if the technique were to be applied, it would be necessary to standardize and evaluate the technique for the particular fiber species and size distribution. In particular, the magnetic separation technique will become less effective as the amount of iron in the fibers becomes less, and for some species of amphibole it probably will not achieve a useful separation.

Magnetic separation cannot be used in its simple form to separate chrysotile fibers from water samples, and other methods must be used. One of the methods investigated incorporated ozone-UV oxidation of the water sample, followed by selective filtration. The method was intended to reduce the turbidity of the water sample, and at the same time to retain the original fiber concentration. The experiments indicated that in some water samples, filtration through a 0.8 μm pore diameter Nuclepore filter reduced the turbidity from 0.109 NTU to about 0.006 NTU. The experiments also showed that the majority of the chrysotile fibers with size distributions similar to those encountered in drinking water would pass through Nuclepore filters of 1.0 - 3.0 μm pore diameter. This, therefore, constitutes a simple and useful technique by which chrysotile fibers can be concentrated relative to the other particulate species present. It is difficult to estimate the concentration factor, because the turbidity is not a direct measure of suspended particle concentration. However, the turbidity was reduced by more than a factor of 10 in most of the experiments, using a procedure that allowed about 50% or more of the fibers to remain in the water. It is likely, therefore, that this corresponded to a concentration factor of about 5, or possibly higher, if the volume of the sample were then reduced either by evaporation or filtration and re-suspension.

The other potential method for separation of chrysotile fibers was based on the observations of Chatfield et al[13], who determined that the inside surfaces of plastic containers could scavenge fibers from suspension. Experiments were carried out to develop this effect as a fiber separation technique. The initial results showed that increasing the surface area of plastic in the containers did not apparently increase the collection efficiency. However, the chrysotile separation efficiencies were very low (less than 20%), leading to the conclusion that the technique was for unknown reasons not working as it had done in the previous studies. It was decided that further investigation of the mechanism was required, but that this could not be accomplished within the constraints of this project, particularly in view of the large concentration factors required if the turbidity measurement was to be capable of detecting chrysotile concentrations of 1 MFL. Unless the detection level of the turbidity instrument could be improved further, detection of 1 MFL of chrysotile by the unmodified instrument would require concentration of the chrysotile from 130 liters of water. The modified instrument, which would yield a fiber-specific measurement, would require concentration of the chrysotile from 1300 liters of water for a detection level of 1 MFL. These concentration factors are clearly impractical because of the large volumes of water required. However, the magnetic alignment - light scattering instrument of Chatfield and Riis[4] would require sample concentration by only a factor of two, which may be achieved easily by the selective filtration technique. Moreover, higher concentration factors would be practical, since the sample volume required is only 5 mL, rather than the 65 mL or 130 mL required for the turbidimeter.

In summary, the modified turbidimeter in combination with magnetic separation, has been demonstrated to provide a simple means of monitoring the concentrations of high iron amphibole fibers in drinking water. This

measurement would be fiber-specific. It is possible that, if a selective filtration step were incorporated, the low-iron amphiboles could also be measured down to about 1 MFL. The detection limits of the instrument, both in its original configuration and in the modified form, were not adequate to provide a realistic method for monitoring of chrysotile at the 1 MFL level.

The magnetic alignment - light scattering system has detection levels which are adequate for direct monitoring of amphiboles at the 1 MFL level. Use of some selective concentration method, however, would be advisable if there were significant levels of other debris in the sample. For chrysotile, the instrumental detection limit of 2 MFL could be improved by use of the selective filtration technique, and values better than 1 MFL could be achieved. The fiber scavenging technique is, however, the more promising separation technique for chrysotile, but a significant effort will be required to define the precise mechanism.

The relevance of the results to studies such as those of Kirmeyer[7], Foley and Missingham[8], Hunsinger et al[9], Lawrence and Zimmermann[10], Hunsinger et al[11,12] and Logsdon and Symons[24] should be carefully considered. In these published studies the turbidities of raw and treated water samples have been compared with their asbestos fiber concentrations, in order to determine if specific standard treatment procedures could reduce the fiber concentrations in treated water in those localities where high fiber concentrations exist in the raw water. In summary, most of the results indicated that if the effluent turbidity were reduced to less than 0.1 NTU, high asbestos fiber concentrations were less likely to occur in the effluent. There is disagreement among these studies as to whether turbidity can be used to monitor the asbestos fiber concentration of the effluent from a water filtration plant. Foley and Missingham[8] state that turbidity measurements can be used as an indicator for asbestos, a conclusion which is contradicted by most of the studies[7,9,10,12,24]. Logsdon and Symons[24], although stating that high amphibole fiber counts are more likely when high effluent turbidities are encountered, found that no relationship appeared to exist between effluent turbidity and chrysotile fiber concentration. They found that at low turbidities, both high and low fiber concentrations were observed. In all of these published studies, the turbidities reported were greater than 0.06 NTU, and it is clear from the results of this project that the asbestos fibers themselves did not contribute significantly to this value. A chrysotile fiber concentration of 20,000 MFL, for example, would yield a turbidity measurement of 0.06 NTU if other turbidity sources were absent.

It is evident that turbidity <u>may</u> be associated with asbestos fiber concentration in some circumstances. If, for example, they are related in the raw water, they may also be related to each other in the treated water. However, this is not necessarily the case in all water systems. The interactions of asbestos fibers with the other suspended particulate, both organic and inorganic, and with the coagulants used in the filtration procedure, are not fully understood. Consequently, since asbestos fibers usually make an insignificant contribution to the turbidity measurement, an assumption of this nature would be unjustified.

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APPENDIX A

SIZE DISTRIBUTIONS OF ASBESTOS FIBER DISPERSIONS

The test dispersions of asbestos fibers used in this study were prepared according to the procedures described by Chatfield, Dillon and Stott[13]. A known weight of the asbestos was dispersed in double-distilled water, taken directly from the condenser of the glass still. The fiber dispersion was packed in clean glass ampoules, after which the tops were flame sealed. The ampoules were then autoclaved for 30 minutes at 121°C to sterilize the contents. The fiber concentration data are shown in Table A1. Three measurements were made on each dispersion and approximately 300 fibers were counted for each determinaton. It can be seen that the mass concentrations computed from the fiber counts are comparable with the theoretical concentrations calculated from the initial weights of asbestos used. Figures A1, A2 and A3 show the length, diameter and aspect ratio distributions respectively.

TABLE A1. RESULTS OF REPLICATE ANALYSES OF REFERENCE ASBESTOS DISPERSIONS

Type of Asbestos	Fiber Concentration, MFL		Estimated Mass Concentration, µg/L	Theoretical Mass Concentration µg/L
	Mean	95% Confidence Interval		
UICC Crocidolite	194	148 - 240	27	50
	227	190 - 264	44	50
	256	181 - 331	34	50
UICC Amosite	43.8	33.5 - 54.0	25	28.5
	32.4	25.4 - 39.3	15	28.5
	49.0	31.1 - 67.0	40	28.5
Union Carbide Chrysotile*	1129	939 - 1319	6.8	5.5
	1047	713 - 1380	4.4	5.5
	1220	1000 - 1440	5.6	5.5

* Original Stock suspension was approximately 10^6 MFL. Analyses are of samples prepared by diluting this 1:1000

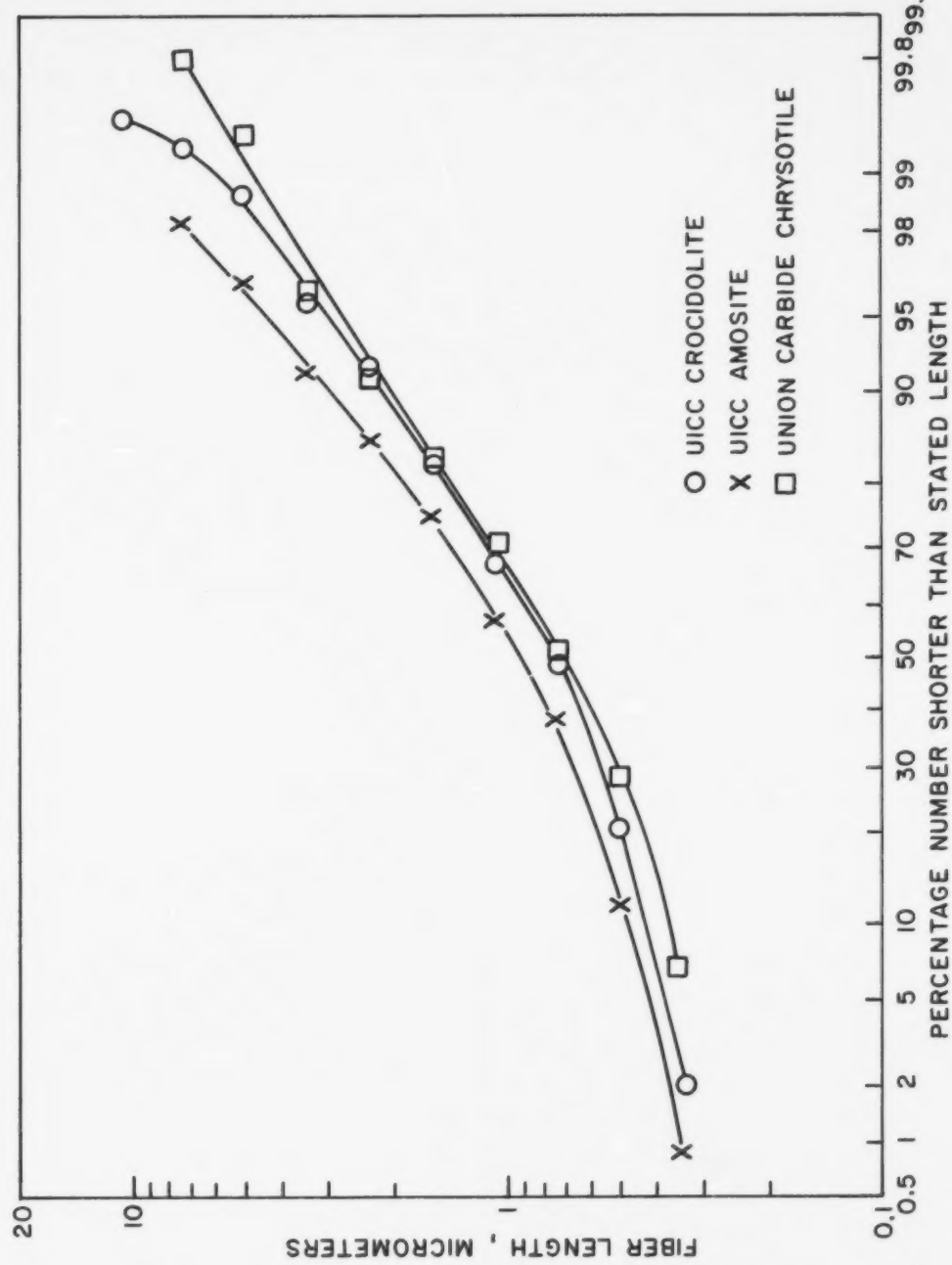


Figure A1. Fiber Length Distribution of Reference Dispersions.

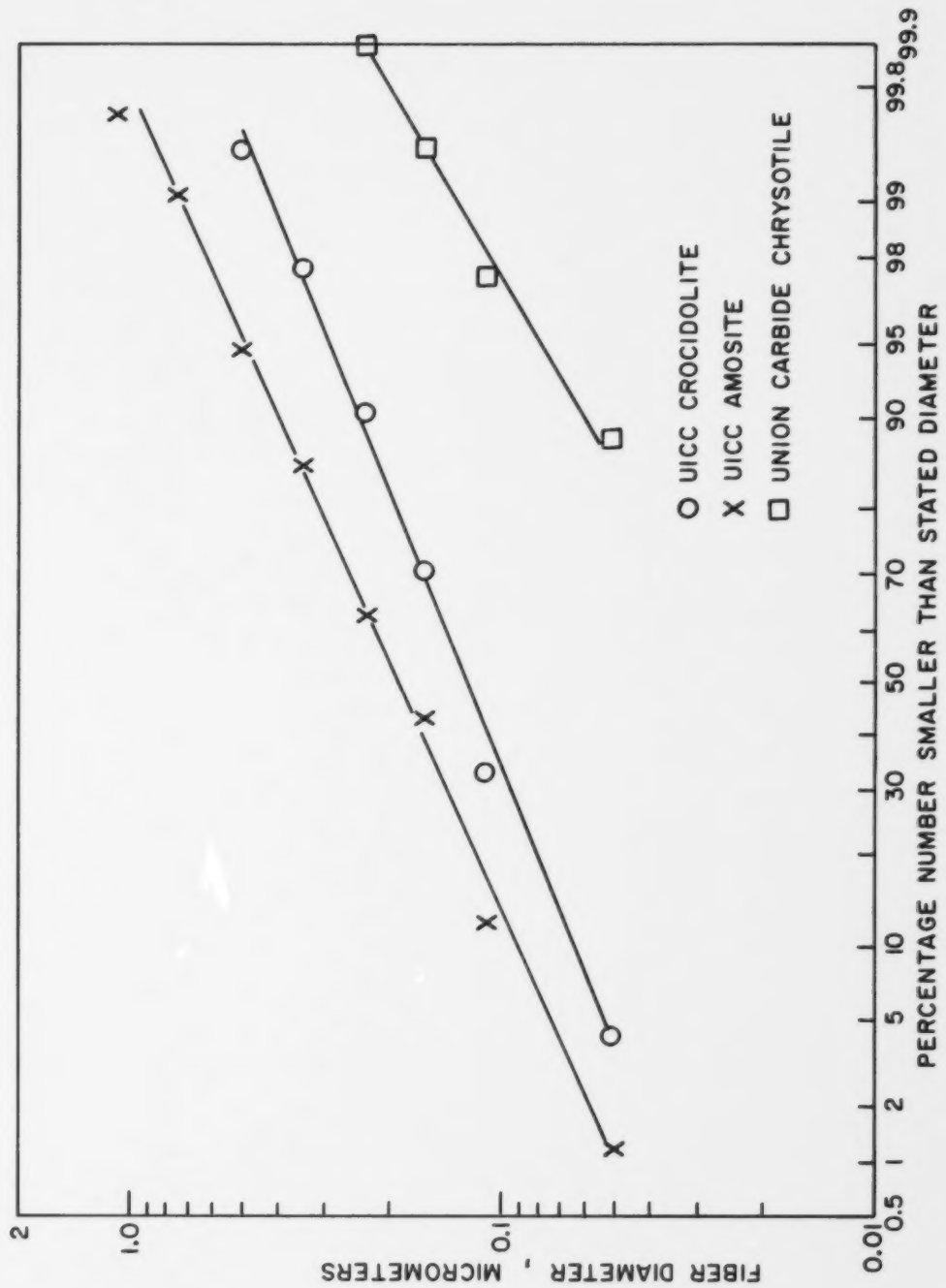


Figure A2. Fiber Diameter Distribution of Reference Dispersions.

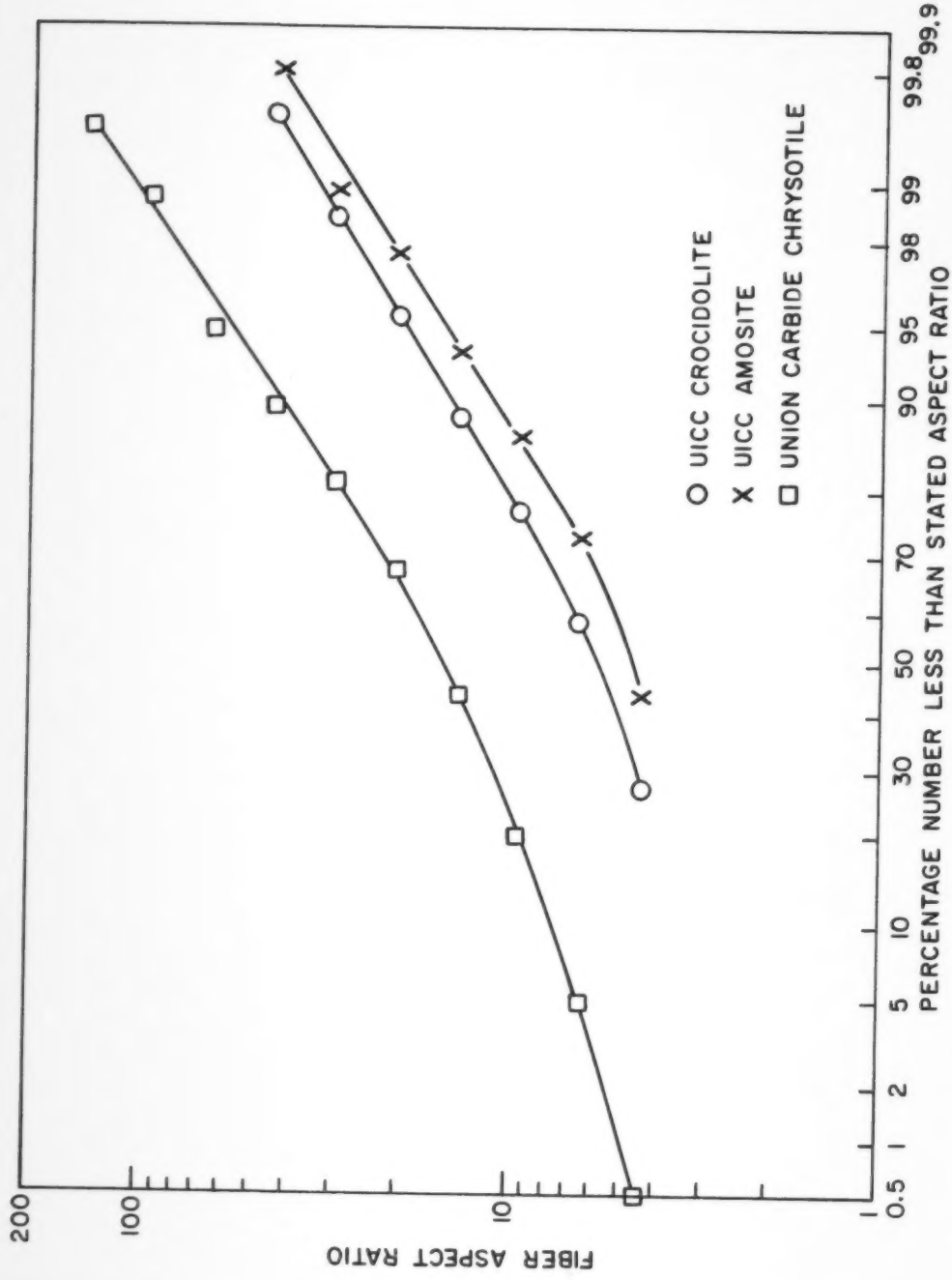


Figure A3. Fiber Aspect Ratio Distribution of Reference Dispersions.

APPENDIX B

EXAMPLES OF SCATTERED LIGHT PROFILES OBTAINED USING THE MAGNETIC ALIGNMENT - LIGHT SCATTERING SYSTEM

The scattered light profiles obtained for various concentrations of UICC crocidolite, UICC amosite and Union Carbide chrysotile are shown in Figures B1, B2 and B3 respectively. It has been found that equant particles which rotate in synchronism with the magnetic field generate a scattered light profile with peaks at 45° and 225° to the field orientation. All such material yields the same profile shape. Before computing the areas of the asbestos scattered light profiles, the contribution to these made by the equant particulate is subtracted. In each of the asbestos scattered light profiles shown, the total signal is represented by the continuous line. The broken line represents the profile which remains after subtraction of the "general particulate" contribution. The areas under the profiles represented by the broken lines are those used in the data analyses.

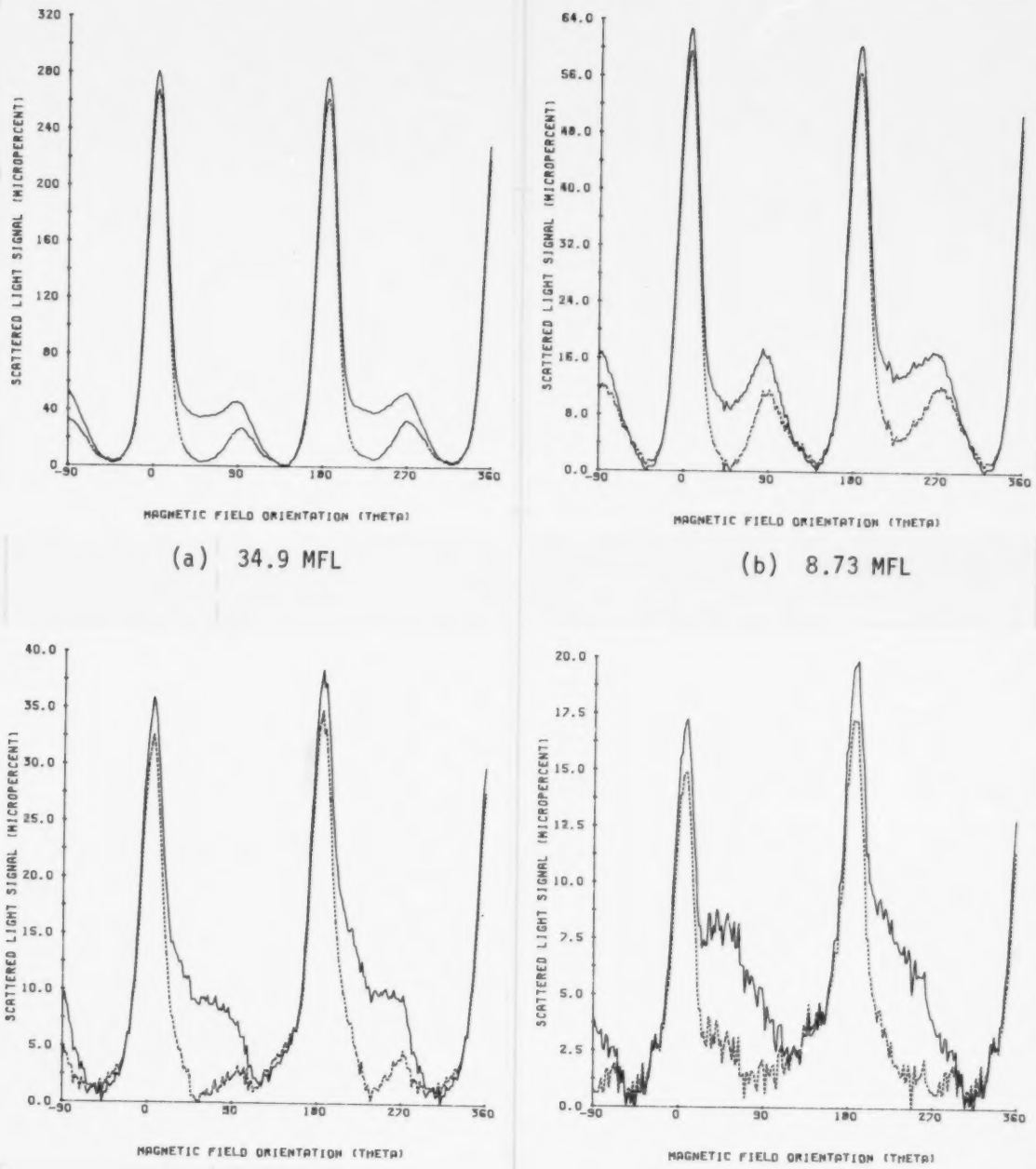


Figure B1. Scattered Light Profiles for UICC Crocidolite.

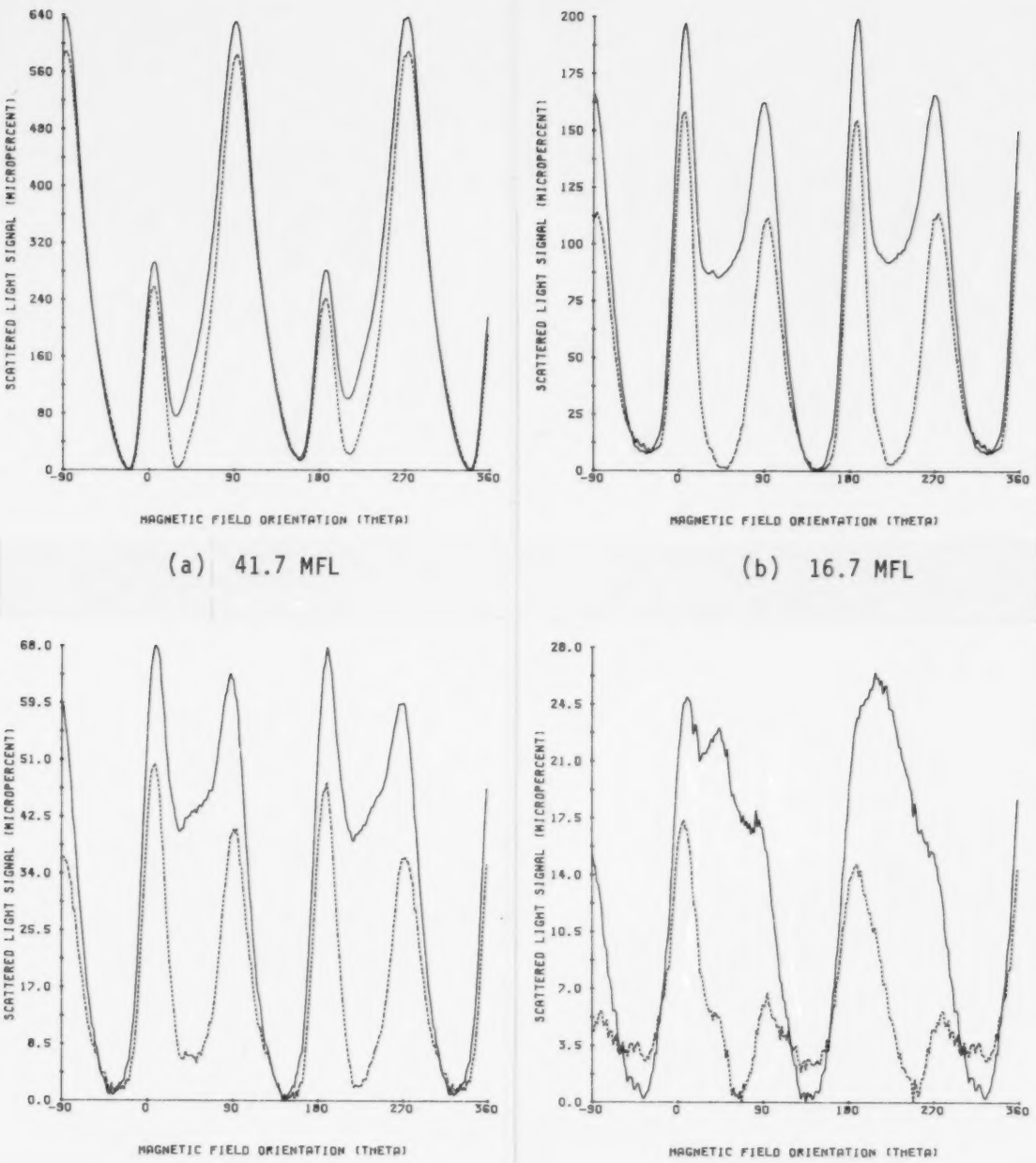
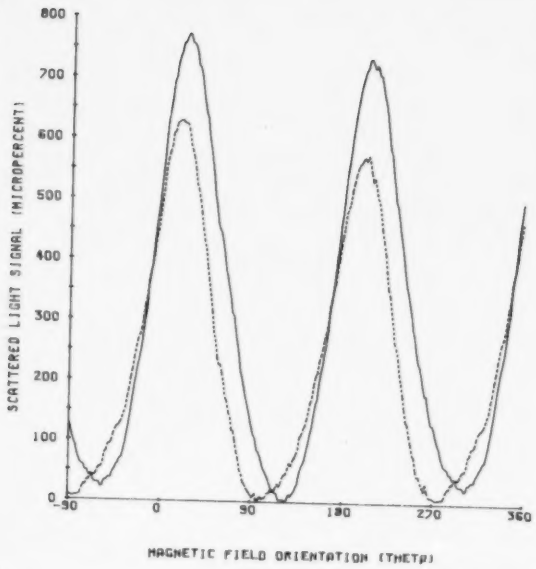
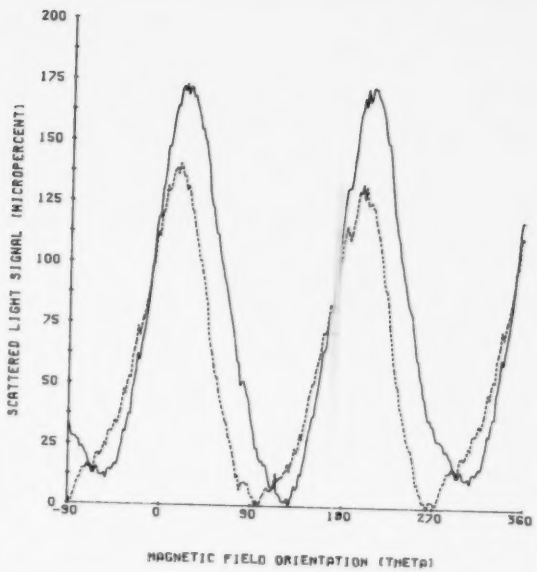


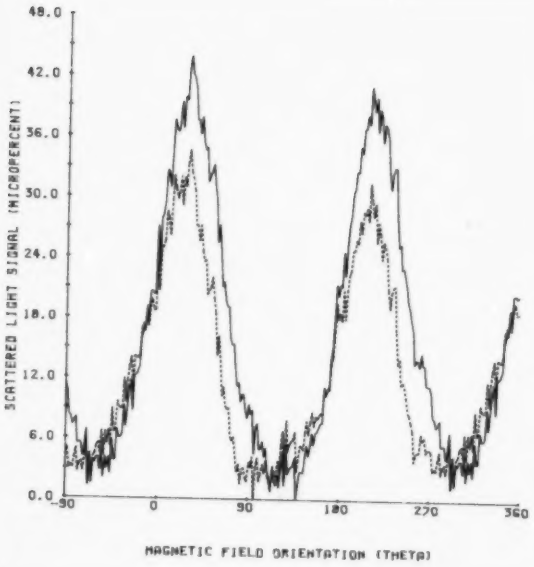
Figure B2. Scattered Light Profiles for UICC Amosite.



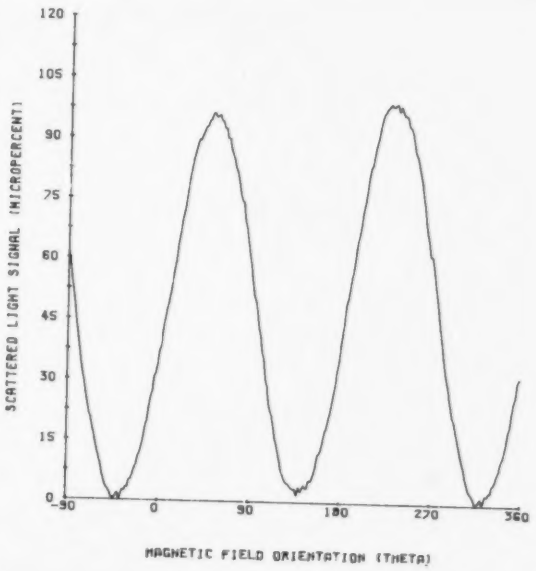
(a) 1000 MFL



(b) 300 MFL



(c) 100 MFL



(d) Ground Glass

Figure B3. Scattered Light Profiles for Union Carbide Chrysotile and Ground Borosilicate Glass.

APPENDIX C

DERIVATION OF CHRYSOTILE FIBER COLLECTION EFFICIENCY CURVES FOR NUCLEPORE FILTERS

A technique originally used by Couchman and Moseley[21] for determination of the stage collection efficiencies of cascade impactors was applied to measure the collection efficiencies of Nuclepore filters. In principle, the method consists of filtration of a challenge dispersion of chrysotile asbestos fibers through two identical Nuclepore filters in sequence. The length distribution of the fibers collected on the second of the two filters is then used to calculate the collection efficiency as a function of fiber length.

The following analysis is taken from Couchman and Moseley's paper:

If the source fiber length distribution is $S(\mu)$, and the distributions of fiber lengths collected on the first and second filters are $N^*(\mu)$ and $N^{**}(\mu)$ respectively, then:

$$N^*(\mu) = S(\mu)\, E(\mu)$$

where $E(\mu)$ is the filtration collection efficiency for fiber length μ. The fiber length distribution on the second filter is given by:

$$N^{**}(\mu) = S(\mu)\,[1 - E(\mu)]E(\mu).$$

The ratio $N^{**}(\mu)/S(\mu)$ rises from zero at the zero efficiency point, rises to a maximum at the 50% point, and then returns to zero at the 100% efficiency point. In practice, there is only a requirement for representative samples of the source and second filter distributions to be measured. If $s(\mu)$and $n^{**}(\mu)$ are the distributions of the representative samples:

$$s(\mu) = f_0 S(\mu)$$

and $$n^{**}(\mu) = f_1 N^{**}(\mu) = f_1 S(\mu)[E(\mu) - E^2(\mu)]$$

where f_0 and f_1 are constants. Then:

$$\frac{n^{**}(\mu)}{s(\mu)} = \frac{f_1}{f_0} \quad [E(\mu) - E^2(\mu)]$$

This ratio is maximum at a value μ_0, where $E(\mu_0) = 0.5$, and the peak value of the ratio is $f_1/4f_0$. The ratio of the quantity $n^{**}(\mu)/s(\mu)$ to its peak value is given by $F(\mu)$, where:

$$F(\mu) = \frac{[n^{**}(\mu)/s(\mu)]}{[n^{**}(\mu_0)/s(\mu_0)]}$$

$$= 4\,[E(\mu_p) - E^2(\mu_p)]$$

$F(\mu)$ can readily be obtained from the experimental fiber counts of the challenge dispersion and the fibers collected on the second filter. $E(\mu_p)$ can then be obtained from the relationship:

$$E(\mu_p) = \tfrac{1}{2}\,[1 \pm (1 - F(\mu))]^{\frac{1}{2}}$$

Figure C1 shows the distribution of fiber lengths in the challenge dispersion. Figures C2 - C5 show the distributions obtained on the second filters, for nominal pore diameters of 0.8 μm, 1.0 μm, 2.0 μm and 3.0 μm respectively. Figure C6 shows the derived filter collection efficiency curves as functions of fiber length.

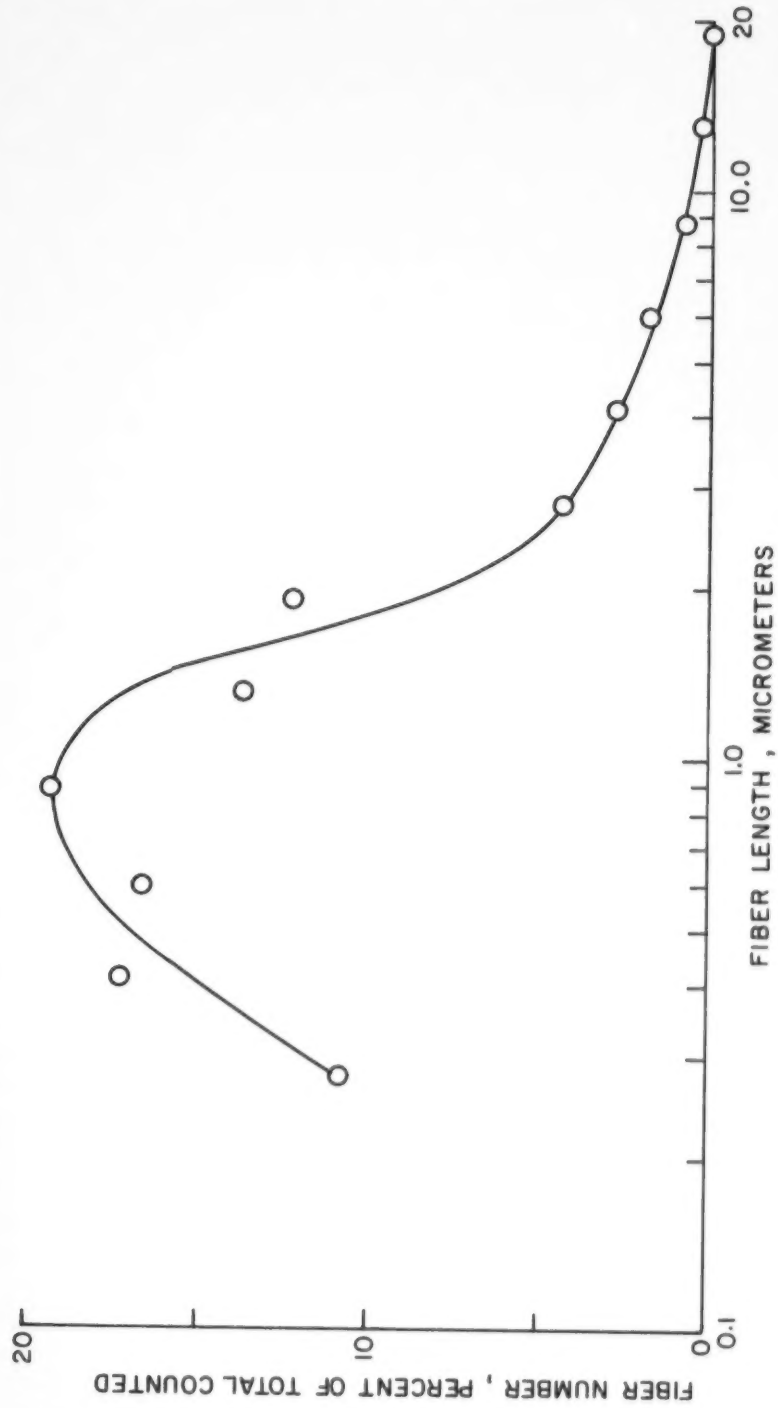


Figure C1. Length Distribution of Filter Challenge Chrysotile Fiber Dispersion.

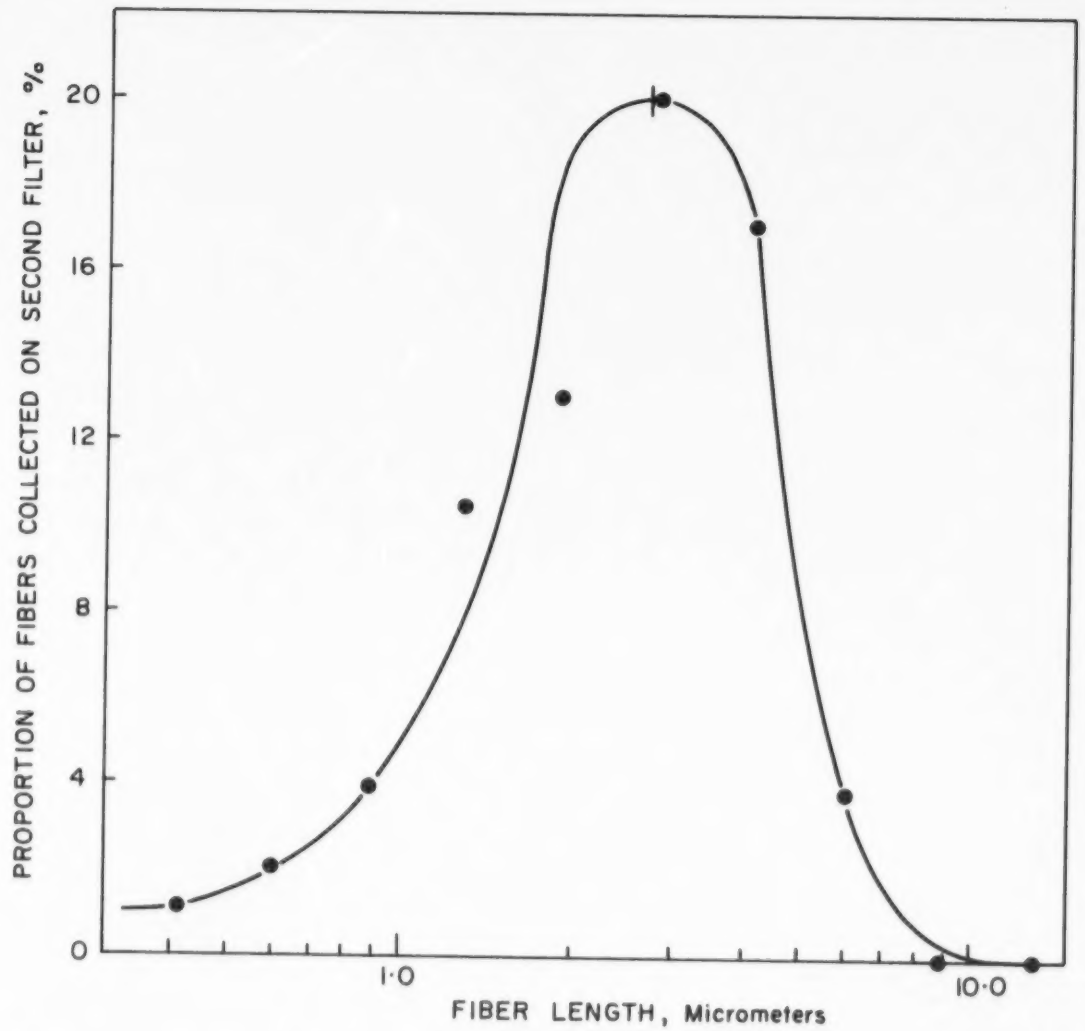


Figure C2. Proportion of Challenge Distribution Fibers Collected on Second Filter, Using 0.8 µm Pore Size Nuclepore Filters in Sequence.

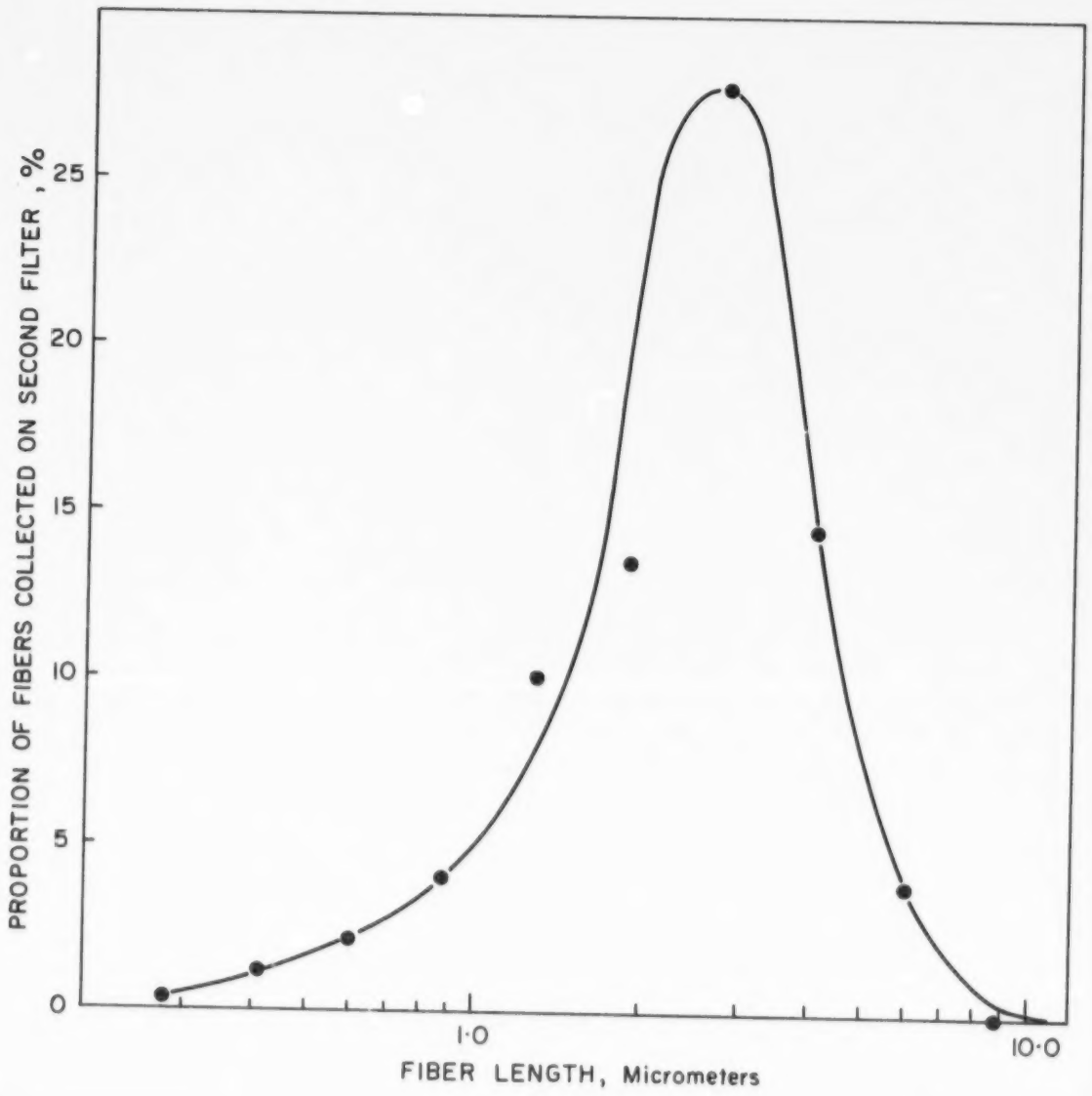


Figure C3. Proportion of Challenge Distribution Fibers Collected on Second Filter, Using 1.0 µm Pore Size Nuclepore Filters in Sequence.

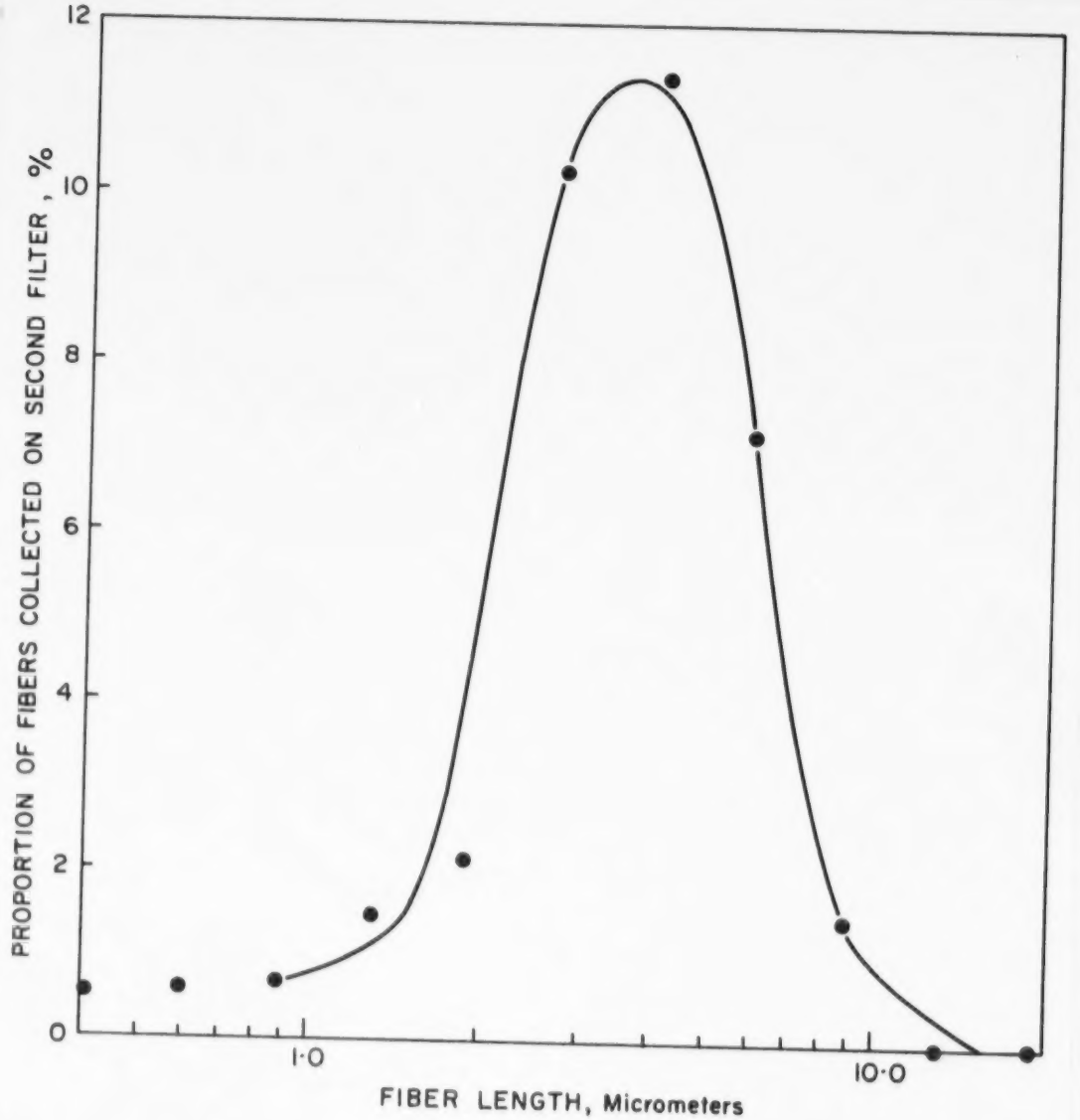


Figure C4. Proportion of Challenge Distribution Fibers Collected on Second Filter, Using 2.0 μm Pore Size Nuclepore Filters in Sequence.

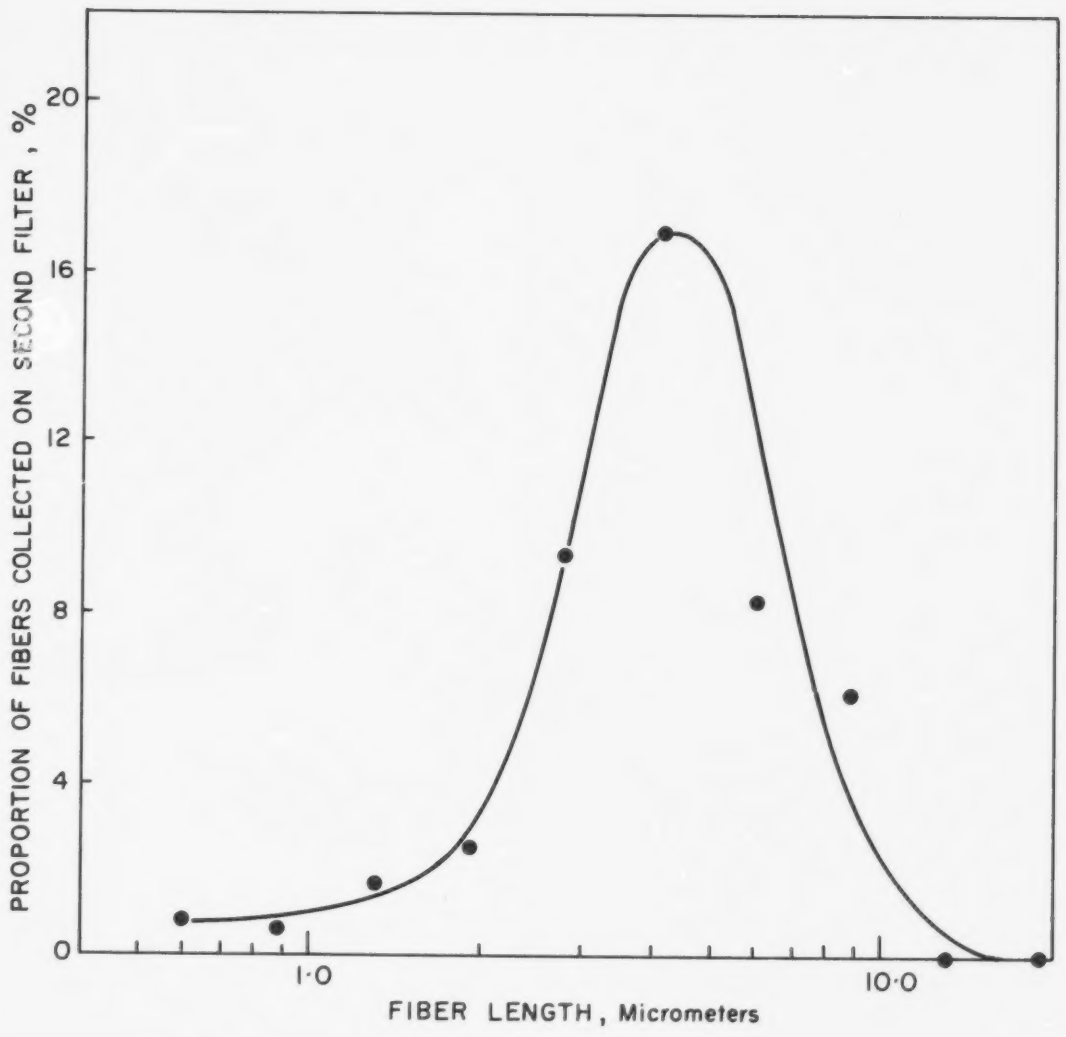


Figure C5. Proportion of Challenge Distribution Fibers Collected on Second Filter, Using 3.0 μm Pore Size Nuclepore Filters in Sequence.

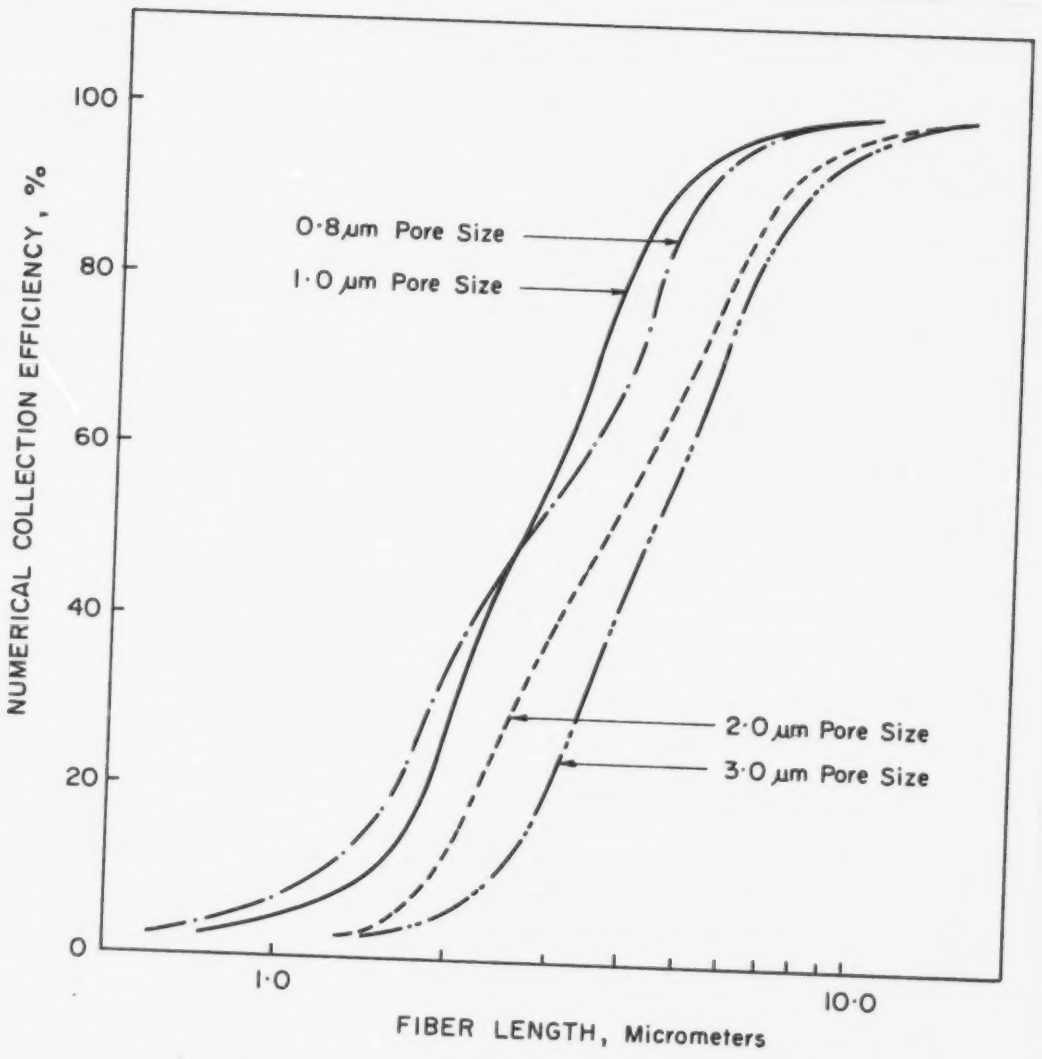


Figure C6. Numerical Collection Efficiency for Single Fibril Chrysotile as a Function of Fiber Length Using 0.8 µm, 1.0 µm, 2.0 µm and 3.0 µm Pore Size Nuclepore Filters (based on counts of approximately 300 fibers).

APPENDIX D

QUALITY ASSURANCE

INTRODUCTION

Since 1976 it has been the policy of the Electron Optics Centre at Ontario Research Foundation to report all relevant analytical parameters in the measurement of asbestos fiber concentrations, and to present individual results as a 95% confidence interval about the mean. Frequent background determinations and blind duplicate measurements are major features of the quality assurance program currently in force in the laboratory.

QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

Since all measurement data will be referenced to results of analyses by transmission electron microscopy (TEM), the quality assurance objectives for the development program cannot realistically exceed those normally associated with the TEM fiber counting technique (Analytical Method for Determination of Asbestos Fibers in Water, EPA Contract 68-03-2717)[3]. These objectives are summarized in Table D1.

SAMPLING PROCEDURES

Collection of water samples was consistent with good laboratory practice with special reference to the requirements detailed in "Analytical Method for Determination of Asbestos Fibers in Water", EPA Contract 68-03-2717[3]. All attachments such as hoses and aerators were removed from the faucets. Faucets were opened and water allowed to run to waste for a sufficient period to ensure that the samples were representative of the current water supply. Samples were collected in pre-washed conventional polyethylene bottles. No preservatives were added. The bottles were labelled and a record made of the details of the collection, including time, operator, location, and the sample number if it was one of a series.

SAMPLE CUSTODY

All samples were collected by the Electron Optics Centre staff, thus ensuring continuity of sample custody.

CALIBRATION PROCEDURES

Calibration of electron microscope magnification is performed once per week, both at fiber counting magnification and grid measurement

TABLE D1

QUALITY ASSURANCE OBJECTIVES

MEASUREMENT PARAMETER	REFERENCE	EXPERIMENTAL CONDITIONS	PRECISION STANDARD DEVIATION*	ACCURACY*	COMPLETENESS
Asbestos Fiber Concentration	New Method Development; Results to be compared to TEM measurements by EPA Method Manual[3]	Drinking Water which meets a turbidity goal of 0.1 NTU	±30%	±50%	±90%

*Accepted precision and accuracy of TEM technique

magnification. Equipment calibration and blank determination on the turbidimeter and the magnetic alignment system was performed both before and after each group of samples was examined, with at least one calibration and blank measurement for every 10 sample measurements. Since this was a development contract, in addition to the standards supplied with the turbidimeter, standards were prepared which were appropriate for this measurement.

ANALYTICAL PROCEDURES

The standard analytical procedure[3] used for the TEM asbestos analyses was that recently developed at the Ontario Research Foundation and submitted under Contract Number 68-03-2717 to the U.S. EPA, Analytical Chemistry Branch, Environmental Research Laboratory, Athens, Georgia.

DATA REDUCTION, VALIDATION AND REPORTING

Data reduction for the TEM asbestos analyses is achieved by a computer program which forms part of the new analytical procedure[3]. The computer program computes 95% confidence intervals, and chi-square tests are made to ensure sample uniformity. All data are checked before and after computer entry by two individuals, prior to submission to the Quality Assurance Officer.

INTERNAL QUALITY CONTROL CHECKS

Measurement of asbestos fiber concentrations has a subjective component, and reliable data can be produced only by using a strict program of replicate and spiked samples, together with frequent blank measurements. Repeat measurements by other operators are also required so that operator bias can be detected and corrected. This Quality Assurance system is already in routine use in the laboratory. Under EPA Contract 68-03-2717, the laboratory developed reference standard asbestos suspensions which are used in the Quality Assurance program.

A stringent program for contamination control is essential in all work on environmental asbestos sampling and analysis. Blank measurements are performed routinely at a rate significantly higher than that specified by the analytical procedure.

PERFORMANCE AND SYSTEM AUDITS

Fiber concentration measurements by TEM have been made on inter-laboratory samples, and the results compared with those from several other laboratories all over the world. These inter-laboratory studies include several conducted by ASTM E-4 Joint Task Groups Sub 11 and 15, and the EPA quality assurance program conducted by the National Bureau of Standards. The project manager has conducted comparisons recently on behalf of the ISO Joint Working Groups ISO/TC147/SC2/WG18 - ISO/TC146/SC3/WG1. In each of these inter-laboratory studies, the results of the analyses performed in the Electron Optics Centre at ORF compared favorably with those obtained by the other laboratories experienced in this type of analysis. No externally

administered routine or formal audits of TEM fiber counts have been established at this time.

On 15 July 1983, the Project Officer visited Ontario Research Foundation to inspect the laboratory and to conduct a quality assurance audit.

The quality assurance audit included inspection of the transmission electron microscopes. The most accurate method currently available for determination of levels of asbestos in water is the transmission electron microscope (TEM) technique developed under Contract 68-03-2717. Therefore, under Contract 68-03-3147, measurements by this TEM technique have been performed to provide reference values for comparison with experimental data obtained using the turbidimeter. Records of the calibration checks and the maintenance schedule for the TEM were examined by the Project Officer. In addition, the Project Officer examined the Sample Preparation Records which itemize the preparation, analysis and results for all samples, associated blanks and background checks.

A number of examples of checks on inter-operator variation were also provided for the Project Officer. It was agreed with the Project Officer that these records and practices would continue to be maintained. The cleanroom facilities for preparation of water samples with known low levels of asbestos were also inspected and found to meet the requirements of this work.